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Inspection in Its Relation to Printing

By MYRON W. ALLING

From the accompanying remarks about inspection, in its relation to the vast number of chances against catching the defects of printing in time to prevent the loss of any chance for profit, there is no question about it being a work for which every mill should receive, in compensation, a price that is in proportion to the risk that is taken, and the great skill that is required in carrying on the work.

ALTHOUGH printing, in print works, has been practiced for two hundred and forty-two years, ninety-five years of this time has been taken up in arriving at the stage of development, where engraved rollers or cylinders have replaced engraved copper plates.

It is unfortunate that rollers were not used right from the beginning because then we would have had two hundred and forty-two years, instead of only one hundred and forty-seven years to practice giving proper attention to preparing the fabric, color, engraving, doctor blades, blankets, lapping, backgreys, and all of the other seen and unseen accessories to printing.

During these one hundred and forty-seven years, there have been so many things outside of the print room, which have influenced the quality of the work, that the confusion has blinded many printers to some of the faults which should have been corrected by them long before the time when, perhaps, some novice could point directly to the trouble with such convincing proof that the question of it being anything but a printing problem was entirely removed.

In a great measure, the development of printing has been hampered by the lack of concentration and general education in regard to the reason for spoiled work.

Bell, the inventor of the printing machine, in a relatively short time, successfully overcame all the difficulties of the problem of inventing and making a printing machine, which has extended its usefulness up to the present day, practically unchanged.

We have, yet, after all these one hundred and forty-seven years to find another mind great enough to bring to the front a set of trained men who are thoroughly educated in all of the operations of running a printing machine, engraving the rollers, mixing the colors, aging or steaming the printed fabric, and developing it properly in the washing.

Until such a department of education is thoroughly established and superintendents, master mechanics, chemists, colorists, printers, etc., become specialists in printing, the simplest fundamental principles of printing will generally continue to be unknown and the skill that is contributed by one man in his work will be lost in the next man.

Seconds in Relation to Closing Lots

One has a trying experience in keeping a print room running smoothly. Some mills do not pay very much attention to the quality of their printing; they are principally interested in production. The margin of profit today is so small that it does not pay them to cut down their production for every little defect.

Of course, if the seconds run much over five per cent, they have to buy some of the goods and resell them as remnants, taking the money to buy new goods of the same construction and reprint sufficient firsts to make the closing of the lot satisfactory to all concerned. If conditions are such that closing of lots quickly is not imperative, this method of procedure is all right, but

where a customer wants his goods in the shortest possible time, this method is a serious handicap to both the customer and the mill, due principally to the liability of a cancellation of the order.

There is no question about it being absolutely necessary to have the inspection as nearly perfect as possible during the printing of fabrics, because even then defects will creep in, spoiling three hundred yards (two and one-half per cent) out of every twelve thousand yards printed.

Points of Inspection

The principal point of inspection is in front of the printing machine, but a great many defects can just as readily be caught on the back end of the machine as the cloth is pleated into a truck, so that the responsibility is divided between the printer and the men on the back of the machines.

Having held a position as superintendent, with the responsibility of the performance of these men's duties holding the quality of the work in the balance, I double checked the printing periodically going from one machine to another at regular intervals according to a schedule sheet which I developed and followed daily. On this sheet was a column for each machine, the names of the

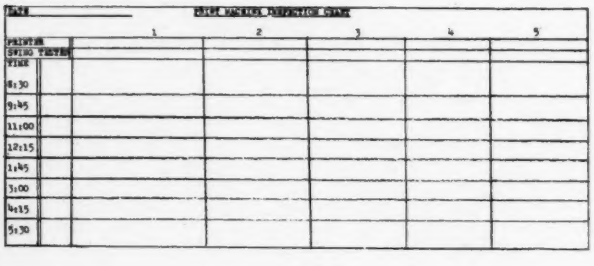

Fig. 1
Inspection chart.

printers and back inspectors, the hours of inspection, and a space for notations concerning any phase of the work, good or bad. At the end of each day I had a complete picture of the day's work, and at the end of each month I was gratified to see the average number of seconds cut down to two and one-half per cent, and in a great many cases very much lower.

It takes a man with a strong conscience to stick to a schedule of this kind, and not be swayed from his duty by the many details of being responsible for the good performance of the work in the many other departments of the mill. Just so long, however, as we put off educating men in a very broad way as specialists in printing, from the preparation of the cloth right straight through to the final development of the printed fabric, superintendents will do well to make sure that their inspection of printing is beyond question.

Of course, there was a very good reason for resorting to a schedule of this kind. The main reason was because the number of seconds was too high, which made closing of lots practically impossible. I tried having an extra printer inspect the printed goods on the back of the machines but found that the very best place for printers to do their inspecting was on the front end of a printing machine, although by going around to the back once in a while they would get an even better idea of the quality of their work.

Practical Illustration of Lack of Inspection

It quite often happens that defects in printing escape the eye of practically everybody in a plant, and in some cases the operators in the dressmaking establishment where it is made up. A member of my family brought home a printed dress which she had purchased at a bargain price. It was a very pretty model, and the workmanship of sewing, to my unskilled eye, may or may not have been perfect. The thing that I was principally interested in, however, was the quality of the printing, so I began to look for some one of the "57" varieties of printing defects that I had been accustomed to look for in my daily work of always being in a pickle. Giving one glance across the front of the dress I noticed one horizontal imprint, and then another, which was foreign to the pattern. These imprints were in the shape of a chain with each link clearly defined.

The printing of cloth is carried on with a backing of endless blanket and unbleached calico, called "backgreys", running on top of it to protect the blanket from getting stained with the color that percolates through the printed fabric. These pieces of unbleached calico sometimes become torn and have to be resewed. If they are resewed on a foot machine with coarse thread, more like string than thread, the bulky seam makes a very great pressure on the printed cloth as it passes between the engraved copper roller and the drum of the printing machine. This pressure causes the color from the engraving to be pushed through the printed cloth to the unbleached calico in back, and makes the print at that point lighter than the surrounding printed area where the color is more on the surface.

The defects which I saw on that dress were due to two of those bulky seams being sewed close together and allowed to pass through the printing machine. No one but a poorly-trained operator would have allowed such a coarse seam to have been put in that cloth which was to provide a smooth and uniform surface, as well as a protective backing for the endless blanket.

Number of Seconds Allowed

There are at least thirty-five outstanding chances for carelessness or oversight by the operator of every printing machine, to say nothing of the many others which are not so noticeable. If any one of these happen to occur while the printer is off his guard and the imperfect printing continues for only one minute, the chances are that at least forty yards will have to be thrown to one side as an imperfect piece or "second" as it is called.

Most customers are very particular about their returns on a lot of cloth that they order in process. There can-

not be too many seconds, and the yards of cloth which they expect to have returned to them must be approximately the same as the number of yards that there were in the grey goods. They do not complain much if the seconds do not run over two and one-half per cent, providing the lot is not short, or some other condition does not make the customer peevish.

A printer is a good one who does not allow himself to miss but a few seconds of time in catching any one of the many defects when they occur. In order to have his day's work a satisfactory one, he has to print on some classes of goods, and under certain circumstances, at least twelve thousand yards of cloth. Of this amount there must not be more than three hundred yards (two and one-half per cent) of imperfect work. Figuring this out in terms of time, his actual running time for printing twelve thousand yards will be in the neighborhood of three hundred minutes (five hours), and the rate of speed will average forty yards per minute. By dividing three hundred by forty, you will find that it only takes seven and one-half minutes out of the day's running time of three hundred minutes to print all the imperfect work that the trade will allow.

There are many other places besides the print room where the cloth may get spoiled. It has to be singed over a red-hot plate or flame, bleached, aged, washed, starched, framed, calendered, cut up in different length pieces, and folded up. In any one of these operations, accidents or carelessness may be the cause of making seconds, so that it is of vital importance to make inspection a most rigid rule in any print room or any of the other departments of a finishing plant.

Importance of Good Bleaching

The principal coloring matters in use today for the printing of cotton and rayon fabrics are Vat colors, Rapid-fast colors, Naphthol colors, Chrome colors, and Aniline Black. These coloring matters print very well on cloth that is bleached and entirely freed from foreign matters. The most sensitive color of all is Aniline Black. It requires special care in bleaching, printing, aging, and washing. It is very susceptible to traces of lime salts left in the goods, so much so, that they entirely prevent the development of the black wherever it happens to fall upon the embedded lime deposit.

These lime deposits cannot be detected in the cloth before printing, or during the printing previous to the oxidation of the color. The first indication of the trouble is sometimes detected on a two or three yard sample of the printed and developed goods, which is taken off from time to time while the cloth is being printed. It can happen that none of these samples will show the defect, because the stains are always deposited in patches at irregular intervals, so that in between, the goods are perfectly printed.

Aniline Black does develop from a colorless print to a black on the hot-drying cylinders in the back frame of the printing machine where the wet print is dried, providing the speed of the machine is not too great, so that a careful inspector would see evidence of the trouble there.

That stage of development of the Aniline Black which has taken place after it has been passed through a fully charged chamber of live steam, is the first real chance that an inspector has to get a view of how well the cloth has been bleached for printing with Aniline Black. By that time two or three thousand yards may have been run in the course of an hour and a large percentage of it spoiled.

A printer really should have sufficient knowledge about bleaching to know that all goods, to be printed with Aniline Black, should be soured off with sulphuric or muriatic acid just previous to the final washing in the bleach house, and to check up with the bleacher, so that he can intelligently go ahead with his printing, and have a reasonable amount of confidence that one of the thirty-five outstanding troubles in printing is eliminated.

None of the other coloring matters in use today give any such trouble and if the cloth is bleached to a good white, is in a state of being practically pure cellulose, and is absorbent, there is no particular trouble, which cannot be seen to guard against.

Removal of Lint

The cloth must be well brushed and wound as straight as possible. The brushing takes away a lot of the lint so that it does not accumulate in the color paste and interfere with the printing.

Straightening Cloth Before Printing

In certain classes of work, due to the weave of the cloth, sheerness of the fabric, the design of the engraved pattern on the copper rollers, and the way it is engraved makes it necessary to either straight-frame or swing-frame the cloth before printing.

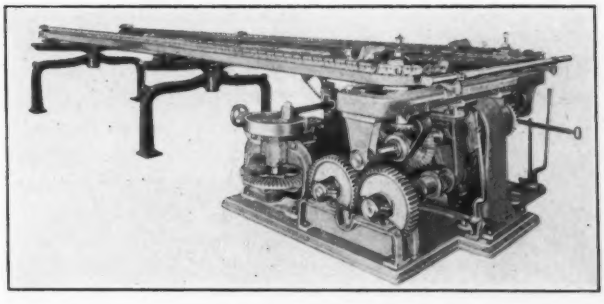

Fig. 2
Straightening cloth before printing.

From the time that the nap on the cloth is as completely burned off as possible on the singer until it is fully bleached in the bleach house, the goods are handled in the rope state. They get many pulls, yanks, and strains which have a tendency to fray, distort, and to get the original alignment of the threads all out of place. This is especially true of lawns, organdies, voiles, broadcloths, filling sateens, fancy weaves, etc. In a great many cases

the print has to actually fit the design on the cloth, even though the design is of the simplest construction, like plain percalines, the print may have to follow exactly the direction of the warp or filling threads. Printed lines in either or both directions should not cross and recross the warp or filling threads as the case may be. Check patterns are really squares and become diamond shape if the cloth is not straight; the cloth must be pulled out to the finished width for printing circles or polka dots—otherwise, they will be egg shape. Handkerchief patterns have to fit exactly the warp and filling threads; otherwise, they will not be square.

If any of these or similar classes of work are printed before the cloth is straightened, the printed pattern will be pulled out of shape when the cloth is finally straightened during the finishing process. It is important, therefore, for a printer to know whether or not the design which he has to print requires framing. A few minutes printing on crooked goods is liable to make a part of his day's work worthless and the whole job unprofitable.

Backgreys

Backgreys, as previously mentioned in relation to the coarse seam showing on the printed dress, must be of uinform thickness throughout. Wrinkles and nips have to be kept out to avoid this trouble of uneven printing due to uneven pressure. Backgreys in poor condition are the cause of much bad printing and many cut outs.

The usual construction of backgreys is forty-eight to fifty inches wide, forty-eight threads to the square inch, running in both the directions of the warp and filling, and two and fifteen one hundredth yards per pound of cloth. The breaking strength, when new, is about sixty in the warp and sixty-nine in the filling. Their life depends upon the kind and amount of coloring matter used, the weight and construction of the cloth to be printed, and the number of times it is used before washing. The thicker the cloth is that has to be printed, the less color strikes through onto the backgrey; the less coverage of color that the pattern calls for, the less there is to get onto the backgrey. Aniline Black and Rapid-fast red have a tendering action on cotton, so that where they are used, the washing of the grey has to be more often. This also holds true where the coverage of the pattern is very great.

The care of the backgrey is of vital importance to the quality of the print on a fabric. If it is run too long before washing, it gets stiff with color paste, becomes tender, rolls up unevenly, with the result that it gets full of holes and breaks. It has to be resewed too close together, and eventually it has to be replaced.

Printers are apt to use them too long before washing, because of the expense of replacing them with new greys, and on account of the expense of washing them. They cannot be washed for much less than five one hundredths of a cent per yard and to replace them, it costs at least fifteen cents per yard. In my experience, I have seen fifyt-eight hundred and fourteen yards (worth eight hundred and seventy-two dollars and ten cents) used up for every million yards of cloth printed.

The best rules for prolonging the life of backgreys is: careful inspection at every step, wash them often, do not run them over scrimp bars, use as little tension as possible, and regulate the speed of the drying cylinders, where they are dried, so that the tension at that point does not tear them apart.

Blankets

As previously mentioned, printing is carried on with a backing of endless blanket and unbleached calico, called "backgreys," running on top of it. This endless blanket is usually about fifteen yards in length, and is made of either all wool, cotton, cotton-piece goods cemented together, or rubber.

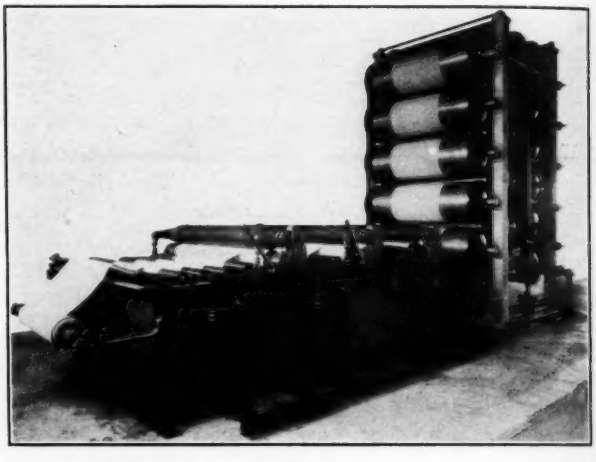

Fig. 3

Back grey washer.

Although it is protected from being dabbed with color to a certain extent by the backgrey, nevertheless a great amount of color gets on the edges, due to the engraved

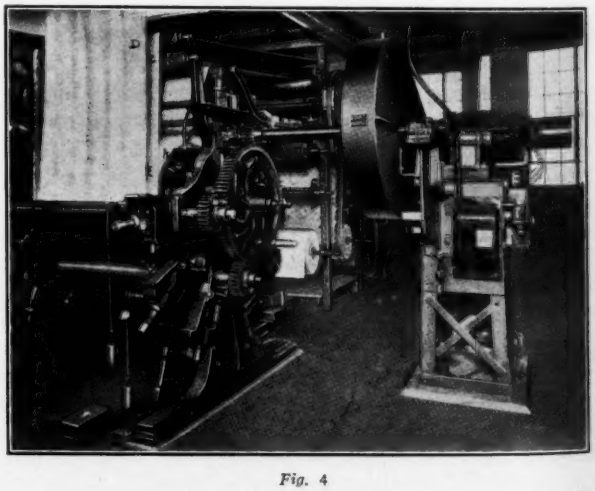

Fig. 4

Blankets

A—Cylinder covered with lapping; B—Blanket; C—Back grey; D—Printed fabric; E—Back grey printed beyond edge of printed fabric (D), striking through and forming an accumulation of color paste on blanket (B).

rollers furnishing color beyond the edges of the cloth which is being printed. It is this color which percolates through the backgrey and gradually builds up and collects in bumps, ridges, etc., on the blanket, that the printer must watch. The printed fabric does not run exactly in the same track all of the time; consequently, it rides on this high-uneven surface, which, due to the extra pressure, will show evidence on the printed cloth, by making that part several shades lighter than the surrounding print. The conditions of the blanket must be watched by the printer, from time to time, to see that it is in good working condition; otherwise, one of the thirty-five outstanding troubles will occur.

Hard foreign substances, such as nails, bolts, nuts, etc., sometimes accidently get on the blanket and cut it, or the printer may be careless and let it get too loose so that it puckers and nips while it passes through the printing machine, thereby cutting through the fabric. Any damages of this sort will show on the printed goods but may not be noticed until it is too late if the inspection is lax.

If a blanket gets its share of inspection, is washed often enough, turned over once in a while, kept free of color, elevated high at the middle of the top strand of the stretch, so that it does not have to be run too tight, it will resist the wear and tear to a marked degree.

Lapping

Underneath the blanket is a padding or lapping, as it is called, which is wrapped several times around a large cast iron cylinder. When the lapping is new it may be put on, all in one piece, but it can be put on in several pieces, providing it is so wrapped that the ends of the pieces do not leave a ridge when the other pieces are wound over them. Of course, every ridge or crease will make an uneven surface and make the pressure greater, so that the print will be affected in exactly the same manner as it was by the coarse seam which was responsible for the chain-like appearance on the printed dress.

Every printing machine cylinder has anywhere from one to sixteen engraved rollers pressing against it, according to the number of the individual colors in the pattern to be printed, or the number of rollers that the printing machine will accommodate. The pressure of these printing rollers against the bowl or cylinder has to be great enough to work on the elasticity of the lapping, blanket, and backgrey in order that the material to be printed shall have the proper spring in back of it to push the cloth into the engraved portions of the copper rollers, which are filled with color paste, and blot out or receive the impression of the color which lies in the engraving.

It takes a good deal of skill on the printer's part to put on this lapping properly. It must be wound good and tight so that it will not slip. After running awhile it stretches and has to be rewound. If this is not done when it becomes necessary, the fabric breaks right across the piece, and oftentimes the break occurs underneath the top layers, so that the only way it can be caught, is by noticing whether or not there is a variation in the printing, due to the uneven surface at the break.

As explained before, a printer has only seven and one-half minutes leeway out of a day's run to spoil goods beyond the point where it will be impossible to give the customer a satisfactory return of his order; therefore, he must catch such troubles immediately upon their occurrence and, too, with the present eighteenth amendment being a binding rule of conduct, there is one less alibi to strengthen a position of being in the wrong.

Mechanism of Printing Machines

Printing machines are constructed so that they print several colors simultaneously. A separate engraved roller is required for each distinct color in the design. Portions of the design are distributed over as many engraved rollers as there are distinct colors in the pattern. There are adequate adjustments on the machine to make it possible for each part of the design to fit or register with that part which it is supposed to join, so that when they are all joined according to the original painted sketch, the pattern is said to be "in fit."

The engraved rollers are all placed in bearings alloted to them so that they are one after the other in contact, under sufficient pressure, with a large central-cushioned cylinder, sufficient to produce the best impression that it is possible to obtain from the color which lies in between, and in some cases over the top of the lines of the engraving.

Under each one of these rollers is fastened a trough containing the color paste and a furnisher roller or brush which rests against the engraved roller, and is partly submerged in the color paste, so that when the printing machine is set in motion, the engraved roller is turned by the frictional contact with the large central-padded cylinder, and in the same way, the furnisher roller or brush is turned by the engraved roller, unless there is a slippage; in that case, it is geared to the mandrel shaft around which the engraved roller is forced in order to furnish it with a temporary axle. This arrangement allows the color paste to be steadily applied in copious quantities to the engraved roller. In back of the engraved roller, about midway between where the cloth to be printed receives its color impression and the point of contact of the furnisher roller, rests the "doctor blade" for cleaning or scraping off all color from the plain surface of the engraved roller.

These parts, the padded central cylinder, the engraved roller, the color box, the furnisher roller, and the doctor blade, constitute the simplest form of a printing machine.

Evidence of Need For Inspection

My story, as you will see, is gradually building up the basis for the need of the closest kind of inspection. So far I have only touched upon watching out for the possible defects in the backgreys, the bleaching, the brushing, and the framing of the cloth, the deleterious result to the backgrey from certain kinds of color, the necessity for clean backgreys, damages to the blanket, the piling

up of dried color on the blanket, the condition of the lapping, the way it is wound and its elasticity. In addition to these eleven vital points of inspection, the matter of the condition of the engraving, the set or pressure of the engraved rollers, the filing and the type of the doctor blade, the working of the furnisher roller, the position of the color trough, the condition and maintenance of the color paste, are of vital importance to inspect, anyone of which, if not properly cared for, will be the means of ruining thousands of yards of goods.

Engraving

Practical printers, as a rule, are not practical engravers; consequently, they can never recognize, to the fullest extent, the limits to the art of printing. They learn by experience something about engraving, but it is very superficial, and besides, it takes years to learn almost anything by experience.

This makes printing very costly to the mill and most difficult for the printer to help himself from getting way beyond the limit of time and yardage for printing bad work.

Some of you have never heard of sinks, misses, shallow engraving, scale of lines in the ground, lines below or up to the top of the surface, sketchiness, and repeat mark damages, in relation to engraving. They are all very important classifications of points in engraving, that necessitate either repairing or returning the rollers to the engraver to be done over.

One of the principal methods of engraving a copper roller is done by etching the pattern with nitric acid. The roller is first evenly coated with a thin film of varnish and then the pattern is cut through the varnish by means of a diamond point, which exposes the copper, so that when the roller is brought into contact with the nitric acid solution, the copper is dissolved out or etched in the exposed parts.

Another method employed for finer work than is obtained by the etching process is by the use of a steel mill, which has upon its face, the pattern to be engraved in relief. This mill is made to revolve against the copper roller with sufficient pressure to indent the pattern into the surface of the roller. The engraving is done a part at a time according to the length of the mill. After engraving the first part, the mill is lifted and the adjoining part is engraved, and so on until the entire length of the roller is engraved. Any variation in the side fit of the impression made by the mill on the roller will show a distinct sketchy appearance on the cloth when it is printed. Such a job turns out perfectly, without question, passable with doubt, as to whether the customer will make a complaint about it or not, or, it shows such poor workmanship that the roller has to be turned off and done all over again.

With the first method, it often happens that the varnish is distributed unevenly on the roller and the varnish is so thin, in spots, that a slight etching takes place at those points. This makes a low place on the roller where the color collects and cannot be entirely removed by the doctor blade, as it scrapes off the color from the plain surface of the roller, so that this faint smear repeats itself every revolution of the copper roller when printing. It is very hard for the printer to notice these smears when printing with certain colors which are not fully developed at the time of printing, so the danger is great, until it is thoroughly established that the engraving is free from defects of this kind. When it is caught, the roller has to be taken out of the machine and polished down with a "Water of Ayr" stone and water, until the entire surface is even with the bottom of the sink. If it is too deep, the roller has to be turned off, and the work of engraving done over.

Misses are parts of the pattern which the engraver has neglected to scratch on the varnished roller. The first print of a new pattern has to be scrutinized very carefully before going ahead, to make sure that nothing is missing.

Shallow engraving is either the result of the saw-teeth corrugations or lines being too far apart in relation to the depth of the engraving or these teeth-like lines are as high at the tips of the surrounding plain surface of the roller, so that the support of the doctor blade at those points is below the surface, which allows the doctor blade to spring or dip into the engraving and scrape out so much of the color that the impression is made weak. This is apt to happen on solid colored dots of rather large dimensions. These lines or grounds should not be engraved any coarser than thirty, or any finer than forty to the inch, and their peaks should extend to the top.

Standardized education for mill men concerning engraving and all other work in connection with printing backed by proper research work to sustain the proven theories in order to stop diversified opinions, and the development of improvements from such a point, would make for real progress as far as the actual quality of printing is concerned.

Accidents are bound to happen to engraved copper rollers. They are liable to be knocked over and bruised, a nail may be driven crookedly into the box in which they are shipped, etc. Any dent will print in the same manner as the indentation made by the engraver. A great many printers know how to fill up these damages with copper dust and smooth up the surface so that they will not show in printing; of course, if the damage is beyond repair, there is nothing left to do but have the roller re-engraved.

Doctor Blades

It does not require a classical and cultural education, or even a practical vocational training, to learn to shave one's face. It does, however, require the greatest amount of knowledge concerning steel to make a razor blade with an edge that will give service and keep the user from

cussing beyond reason, for its quality depends not only upon the texture of the skin to be shaved, but also upon the thickness and hardness of the razor blade, the angle at which it rests against the face, the pressure applied to it, the kind of shaving soap used, and the stiffness of the beard which it has to clean off.

In exactly the same way, this simile or analogy can be applied to the practical handling of the doctor blade, only in place of cleaning whiskers, it has to clean off color paste from the face of an engraved copper roller, the texture of the face varying according to the weight and scale of the engraving and the stiffness of the material to be cleaned varying according to the viscosity and tenacity of the color paste. Every man shaves in a different way, and every printer has his own ideas how to obtain the best results from his doctor blade, in regard to edge, placement, angle, gauge, pressure, and thickness of color.

It is not an easy matter to hone a razor, and neither is it an easy matter to put an edge on a doctor blade that is suitable for different kinds of engraving, as it requires unerring judgment and long practice for its proper performance.

There are many defects in printing caused by the conditions surrounding the doctor blade. One little piece of sand in the color will carry up to the engraved roller at the point of contact of the doctor blade, nick it, and possibly scratch the copper roller at the same time; in either or both cases the color that passes through will print a streak on the cloth which makes it useless.

It often happens that as the doctor blade traverses back and forth parallel to the axis of the roller, it hits the side of the frame of the printing machine, so that the blade springs back and forth against the roller, allowing a fine film of color to print the cloth. Such a series of smears is called a "chatter mark." If the tension of the blade against the roller is uneven, chattering is very apt to take place.

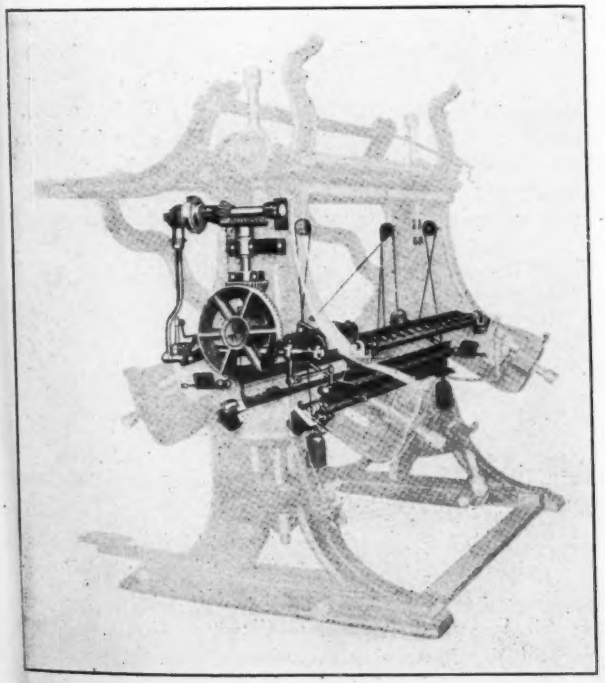

Fig. 5
Doctor blades.

Small particles of steel filings from the doctor blade sometimes get into the color and are driven into the engraved roller by the edge of the doctor blade, which lifts the doctor up every revolution of the roller, allowing the color to pass through and print the goods. These marks are called "steels."

Lint from the cloth accumulates in the color trough and gets wedged underneath the doctor, which also lifts it up enough so that it can get through, the color that goes with it while it is working its way through makes marks called "snaps."

The tenacity of some colors makes it almost impossible for the doctor blade to remove every last bit of the color from the plain surface of the roller, so that a very thin film of color is printed all over the cloth which makes the effect called "tarnishing."

List of Printing Troubles

If great care is not used in regulating the proper performance of the doctor blade—just as in shaving with a razor—the face of the roller will be cut, and the goods will be printed by the color which is lodged in the cut.

When I started this article I thought that I would stick to a list of only thirty-five outstanding printing troubles as previously mentioned, although I did cover myself by slipping in the possibility of there being "57" varieties. I find that I have used up the alloted number, and have many more left over, however, to satisfy any curiosity that may have been aroused, about exactly what these were, I am going to include in this article my complete list regardless of whether I have written about them or not. They are as follows:

1. Broken blankets.
2. Broken lapping.
3. Chatter marks.
4. Cloth nips.
5. Color specks in the printed areas.
6. Creases or double edges in the cloth on the printing machine, in the ager, and on the soaper.
7. Creases, double edges, or holes in the backgreys.
8. Damages on the washer.
9. Dirt on the cloth.
10. Dirty or lumpy blankets.
11. Drag marks from ager and washer.
12. Doctor streaks.
13. Etching of Aniline Black on succeeding rollers.
14. Insufficient aging.
15. Leaky cans.
16. Mark off's from blanket, ager, print cans and washer.

17. Miscolor.
18. Misfit.
19. One color working into another.
20. Poor bleaching.
21. Resist stains.
22. Scabby printing.
23. Seam marks from backgreys.
24. Short pieces.
25. Sinks.
26. Snaps.
27. Steels.
28. Shaded printing.
29. Sticking in.
30. Stops of Aniline Black and Rapid-fast colors on print-drying cans.
31. Tailing down.
32. Tarnishing.
33. Tender cloth.
34. Water spots from ager and print-drying cans.
35. Wrinkles in cloth due to puckered seams.

PRESENT CRITICAL SITUATION AND ITS REMEDY

Education of men for the practical vocational training of printing, discoveries of new and cheaper dyestuffs, and new inventions of printing machinery have not kept pace with the low prices and the high quality work demanded by the converters. Textile machinery is closed down, the future of the New York textile center is threatened by an unheard of bad market, finishing companies are failing, mill men are figuring which is the cheaper—to close down or operate at a loss—and modern efficiency methods are inadequate to cope with the situation. There seems nothing left to do but for all mills to organize, forget their own advantages of efficiency over their competitors, cease using the low price tomahawk, regulate the number of working hours, so that the output will meet the demands of the consumer, and raise prices to a level that will keep our finishing plants from going out of business.

NEW PATENTS

(*Abstracted by Synthetic Organic Chemical Mfrs. Ass'n*)

Production of Ortho-Amino-Halogen-Anthraquinones. (Relates to the production of dyestuffs and intermediates, especially 1-amino-2-chloranthraquinone in a purified form by mixing together anthraquinone-1-amino-2-sulphonic acid and potassium per sulphate in sulphuric acid, mixing the product with hydrochloric acid and sodium chlorate in an aqueous solution and mixing the resultant product with sodium hydrosulphide.) Rhys Jenkin Loveluck, Ernest George Beckett, and John Thomas, of Grangemouth, Scotland, assignors to Scottish Dyes, Ltd., of Grangemouth, Scotland, No. 1,858,334, May 17, 1932.

Process for Manufacturing Ar-Tetrahydronaphthols and Their Esters. (Dyestuff intermediates and pharmaceutical products, comprises treating naphthol esters with hydrogen in the presence of a base metal hydrogenation catalyst.) Ernst Korten, of Fechenheim, near Frankfort-on-the-Main, Ger., assignor to I. G. Farbenindustrie Aktiengesellschaft, of Frankfort-on-the-Main, Ger., No. 1,858,627, May 17, 1932.

Water Insoluble Azo-Dyestuffs and Process of Making Same. (Obtainable by combining in substance or on a substratum, particularly on the fiber, any diazo-, tetrazo- or diazoazocompound with a 2.3-hydroxy-naphthoyl-4-amino-1-alkoxy-methylbenzene; for use in the production of valuable color lakes and when produced on the fiber yield fast dyeings of various shades and printings.) Leopold Laska and Arthur Zitscher, of Offenbach-on-the-Main, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,858,628, May 17, 1932.

Water Insoluble Azo-Dyestuffs. (See above Patent No. 1,858,628.) Leopold Laska and Arthur Zitscher, of Offenbach-on-the-Main, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,858,629, May 17, 1932.

Stable Preparations of Vat Dyestuffs and Process for Making Same. (Comprise a vat dyestuff, alkali starch, hydrosulfite and monopol soap.) Karl Fritz Maennchen and Frank Hummerich, of Hochst-on-the-Main, Ger., assignors to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,858,632, May 17, 1932.

Azo Dyestuffs Derived from Amino-Ethers of Hydroxydiaryls. (Particularly, the azo dyes of the so-called ice-color or direct-developed type, dyeing cotton and other fibers fast, clear, deep rose-red shades.) Ernest F. Grether and Gerald H. Coleman, of Midland, Mich., assignors to the Dow Chemical Co., of Midland, Mich., No. 1.858,928, May 17, 1932.

Halogen Containing Dyestuffs of the Dibenzanthrone Series and Process of Making Same. (Obtained by the action of formaldehyde in presence of chlorosulfonic acid on such a dibenzanthrone derivative which contains at least one *X*-H group, *X* being O or NH, dyeing cotton, according to the nature of the starting material, blue, grey or red-brown tints.) Karl Krauer, of Basel, Switzerland, assignor to the Firm of Society of Chemical Industry in Basle, of Basel, Switzerland, No. 1,859,146, May 17, 1932.

Manufacture of a Dyestuff of the Anthraquinone Series. (Of a high degree of purity, by treating the blue-green dyestuff described in U. S. Patent No. 1,044,797 with a reducing agent in presence of an alkali, removing the leuco-compound thus separated from the solution containing the secondary dyestuff.) Wilhelm Moser, of Riehen, near Basel, Switzerland, assignor to the firm of Society of Chemical Industry in Basle, of Basel, Switzerland, No. 1,859,151, May 17, 1932.

Vat Dyestuff of the Dibenzanthrone Series and Process of Making Same. (Obtained by treating Bz_1:Bz_1'-dibenzanthronyl with a nitrating agent and fusing the product thus obtained with a caustic alkali; dye cotton fast blue-grey tints.) Bertram Mayer, Hugo Siebenburger, and Karl Krauer, of Basel, Switzerland, assignors to Society

(Continued on page 478)

Design Copyright for Textiles Only

By **WALDON FAWCETT**

To the extent of the interdependence and corelationship of the various processes that contribute to textile production must there be interest throughout the industry in the latest development in the protracted movement to obtain "security" for textile designs. Even if there be chemists who have considered it a far cry from the establishment of monopolies in creative textile designs to the processes of dyeing, bleaching, and finishing, yet is there a challenge to their sense of contact in the most recent measure submitted to Congress by textile interests.

IN a recent measure submitted to Congress it is proposed that textiles alone, of all commodities, shall be singled out as the subjects of a new system of Federal certification destined to discourage imitation and infringement of pattern or color disposition. To thus accord a Governmental facility for the entrenchment of business good will to a lone line of manufactures is an innovation without precedent. But, for all those connected with the textile industry, the especial significance lies not in the special privilege as such but in the circumstance that the very narrowness of the scope or range of the proposed system of registration renders it possible to plan an agency of design authentication peculiarly and specifically adapted to the textile environment.

This concentration disposes, as it happens, of one of the growing drawbacks which, in recent years, has all but chilled the enthusiasm of some textile leaders for national legislation to enforce respect for original fabric designs. When the campaign for design protective legislation was first launched in Congress, a number of years ago, it was a group of textile producers,—notably members of the silk section,—who supplied the primary inspiration. The early editions of the Design Registration Bill, as sponsored by the old Design Registration League, were phrased in forms eminently suitable to textiles, although intended, of course, to be applicable to all the classes of manufactures which depend for their saleability upon decorative or ornamental design.

As one Congress after another considered the design protective program, but never with Senate and House in accord for definite action, the Bill began, gradually, to lose many of the features that had endeared it to textile and associated interests. Being a general bill, applicable to many lines of industry, it drew the fire of criticism from many quarters and as one and then another band of objectors became vociferous it became necessary to offer concessions and make changes in the provisions of the Bill. Some of these mutilations were forced by interests that had nothing whatever in common with textile producers. For example, the modifications insisted upon by the manufacturers of spare parts or repair parts of machinery, stoves, etc.

So serious were the amputations and sacrifices that many erstwhile enthusiasts in textile circles all but lost interest, a few years ago, in the quest for design protection. Prominent members of the industry went so far as to say publicly that they feared that the emasculated Bill offered nothing that could be relied upon to accomplish the paramount purpose,—viz., to stimulate ingenuity in design by insuring rewards for originality. Out of this welter of concession and compromise there now emerges a situation which must make the erstwhile state of conflict a blessing in disguise.

Suspicion came to a head in Congress last year that it would never be possible to reconcile all industries to one system of design protection. Indeed, oddly enough, there was stiff opposition in some quarters to any recognition of novelty in design beyond that allowed by the existing patent system. The only solution appeared to lie in a system of Design Copyright, Limited,—design registration restricted to the industries that believed in and wanted a Governmentally-administered agency for attesting priority of discovery and adoption of designs. As the expression of that sentiment in Congress, the Senate Committee on Patents evolved the draft of a design law limited in its application to five major lines of industry, of which textiles was one. The Bill, which contemplated this trial of a system that might ultimately become universal, failed to reach a vote owing to the filibuster in the Senate.

The new Congress faced a continuance of the differences of opinion among design-users. Agitation for legislation had lapsed even in some of the five fields selected for preferment a year ago. But the die-hards among the textile forces continued to appeal for some system adapted to the pedigreeing of seasonal designs. In the end, their persistence has been rewarded in that the Chairman of the House Committee on Patents has introduced a Bill which contemplates the setting up of machinery for the registration at Washington of designs employed in or expressed in textiles.

Two desirable consequences result from the change of objective whereby the effort for design protection is focused sharply on the textile industry. In the first place, it has been possible to lay out a plan of protec-

tion adapted intimately to the textile line. In the second place, it has been practicable in this one—industry—formula to insert special provisions calculated to allay the one element of opposition which has beset the textile program from the outset,—i.e. the resistence of the organized retailers who have demanded that any institution of design registration shall condone unintentional and unconscious infringement on the part of merchants who handle imitative designs in ignorance of the transgression upon copyrighted designs.

The latest move to provide more elastic, less expensive, and more quickly available protection for textile designs has enlisted throughout the preliminary stages, the active support of a number of leaders in the processing branches of the industry. For example, Mr. Robert Blum, Assistant Treasurer of the United Piece Dye Works aided materially to spur the House Patent Committee to action when he brought to the notice of the Committee members the serious financial consequences likely to result from continued disregard of property rights in original design. He pointed out, that, today, design piracy is imperiling most of the more stable houses in the textile field,—the manufacturers who have the largest plant investments and who employ the largest number of workers. Similarly, in the conferences at Washington, Mr. Charles P. Moss of the Design Protection Association emphasized strongly the aggravation of conditions which followed the abrogation of the Printers' Association agreement on March 27, last. In this same connection, Mr. Moss told the Congressional group that, today, American design is as good or better than French design because "our American designers understand the American woman's requirements and desire for color combinations and color harmony which are divergent from the foreign in many cases."

A design protective bill, fitted exclusively to the textile industry, has,—as demonstrated in H. R. 12528,—opportunity to cope the better with what has been from the outset one of the problems of textile design certification, namely the obligation laid by the current copyright statute, to affix a copyright notice to each repetition of a design. Here, obviously, is an angle of the physical features of design copyright which concerns dyers, bleachers, finishers, etc. if they be called upon to provide for or preserve the legibility of the markings. Section 6 of the Sirovich Bill provides that the letter "R" in a circle shall constitute a sufficient declaration of registration and that it shall not be necessary to so mark each occurrence of the design but a single marking on each ordinary unit of sale shall be sufficient.

After all, though, the one most notable advance exemplified by the Bill for textiles only, over all previous proposals for design protection, is in the enlistment of Federal support for the confirmed claims of a design owner to originality and novelty in his woven or applied design. All previous versions of the proposal for Design Copyright or Design Registration contemplated, on the part of the Government, simply an enrollment of the sworn date of design creation and use. In other words, Federal authority would testify via birth certificate to the beginnings of a design career. Beyond that the agency for entry would assume no responsibility. If any contest or controversy arose in respect to priority or any other issue, the disputants would be called upon to fight it out in court on such evidence as they could assemble. In contrast to this the Bill for the Protection of Textiles provides that the Register shall upon receipt of an application for registration of a design cause a "search" to be made to determine the originality and novelty of the design before issuing a certificate of registration. Thus, under this plan, a Copyright certificate would stand in Court and in trade circles not as a mere dating device but as an authoritative witness qualified to validate priority and at the same time establish a presumption of originality and novelty.

NEW PATENTS

(Continued from page 476)

of Chemical Industry in Basle, of Basel, Switzerland, No. 1,859,212, May 17, 1932.

Manufacture of a Dyestuff of the Anthraquinone Series. (See above Patent No. 1,859,151.) Wilhelm Moser, of Riehen, near Basel, Switzerland, assignor to the firm of Society of Chemical Industry in Basle, of Basel, Switzerland, No. 1,859,255, May 17, 1932.

Dyestuffs of the Urea-Azine Series and Process of Preparing the Same. (Dye wool, cotton and regenerated cellulose materials blue shades varying with the specific sulphonated azine nucleus present in the complex urea.) William Arthur Silvester, of Blackley, Manchester, Eng., assignor to British Dyestuffs Corp., Ltd., of Manchester, England, No. 1,859,477, May 24, 1932.

Compounds Having an Affinity for Cotton. (Comprises treating 1.4-di-(4'-nitrobenzoylamino) benzene-2-carboxylic acid in the presence of an excess of a diluted sodium hydroxide solution with anhydrous sodium sulfide; dyes cotton bright yellow and when diazotized on the fiber and coupled with 1-phenyl-3-methyl-5-pyrazolone yields a strong greenish-yellow, fast to washing and light.) Werner Lange, of Dessau-Ziebigk in Anhalt, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,859,705, May 24, 1932.

Manufacture of Azo-Dyes. (Particularly direct dyes for cotton and pigments fast to light and washing, obtained by subjecting a nitro-benzoylated azo dye to an alkaline reduction; yield shades of golden-yellow, bluish-red, red-violet, and yellow-brown.) Werner Lange, of Dessau-Ziebigk in Anhalt, Ger., assignor to General Aniline Wks., Inc., of N. Y., N. Y., No. 1,859,706, May 24, 1932.

Analytical Methods for a Textile Laboratory—Continued

By **WALTER M. SCOTT, Ph.D.**

Consulting Chemist with Gustavus J. Esselen, 73 Newbury St., Boston, Mass.

(*Continued from page* 460, *July* 18*th issue*)

PART III—METHODS OF ANALYSIS

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INTRODUCTION

The Research Committee of the American Association of Textile Chemists and Colorists has recently appointed a special sub-committee to standardize the analytical methods which are applied to the common chemicals and allied materials used by the textile industry. This action is indicative of the active interest in this particular subject among textile chemists at the present time. Therefore it seems to be an opportune moment to present a collection of such analytical methods which might serve as a basis for discussion in connection with the work of the above mentioned sub-committee. Dr. Scott has had an unusual opportunity to test the value of the methods which he proposes, during his ten years of service as Chief Chemist for Cheney Brothers, silk manufacturers. As an active member of the Research Committee of the A. A. T. C. C., he has kept in close touch with all recent developments along this line.

Description of Methods

No. 1 ACID—ACETIC

$HC_2H_3O_2$

Specific Gravity

Determine by hydrometer at 15° C.

Acidity

Weigh accurately in a glass stoppered weighing bottle about 0.2 gms. of the sample. Wash into a beaker, dilute with distilled water, and titrate with N/10 NaOH, using Phenolphthalein as an indicator. Calculate as $HC_2H_3O_2$. 1 cc. N/10 NaOH = 0.006 gms. $HC_2H_3O_2$.

Residue

Weigh about 10 gms. into a platinum evaporating dish and evaporate to dryness on the steam bath. Cool and weigh, then ignite at low red heat for several minutes, cool and weigh again. The second weight represents the inorganic residue and the difference between the second and first weights, the organic residue.

Qualitative Tests

Test as usual for hydrochloric and sulfuric acids with silver nitrate and barium chloride respectively.

Average Analysis of Commercial Acetic Acid 28%

Specific Gravity	1.0388
Acidity, as $HC_2H_3O_2$	28.0%
Organic Residue	Not over 0.03%
Inorganic Residue	Not over 0.04%
Hydrochloric Acid	Not more than a trace
Sulfuric Acid	Not more than a trace
Color	Clear and Colorless

No. 2 ACID—CRUDE CARBOLIC

(Cresylic Acid)

Chiefly a mixture of C_6H_5OH and $CH_3C_6H_4OH$

Specific Gravity

Determine by hydrometer at 15° C.

Soda Test

25 cc. of the sample are shaken with 75 cc. of 10% sodium hydroxide. Everything should dissolve.

Fractional Distillation

Into a distilling flask of about 250 cc. capacity pour 100 cc. of the sample to be tested. Insert a delicate thermometer so that its bulb reaches to within ⅜ of an inch of the bottom of the flask. The outlet from the flask should lead into a condenser about 2 feet long through which cold water is freely circulating. The flask should be supported by an asbestos board in which a hole has been cut so that the Bunsen flame may play directly on the glass. The distillate should be caught in a 100 cc. graduated cylinder. The successive temperatures at which the first drop, 5 cc., 10 cc., 20 cc., 30 cc., etc., pass over are carefully noted and the distillation is prolonged until 90 cc. have entered the graduate.

Average Analysis

Specific Gravity	1.035
Soda Test	Clear or only slightly cloudy solution
Fractional Distillation	90% by volume distills under 215° C.

No. 3 ACID—FORMIC

HCOOH

Acidity

Make up a solution containing 10 gms. of the sample in exactly 1000 cc. Place a 200 cc. aliquot of this solution in a flask, add Phenolphthalein and titrate with N/1 NaOH until the pink coloration becomes permanent. Calculate as HCOOH. 1 cc. N/1 NaOH = .046 gms. HCOOH.

Reducing Power

Place a 20 cc. aliquot of the above solution in a flask, add 0.5 gms. Na_2CO_3 and 75 cc. water. Heat the mixture to boiling and titrate at this temperature with N/2 $KMnO_4$ until the clear solution above the brown precipitate of MnO_2 is just pink. Calculate as HCOOH. 1 cc. N/2 $KMnO_4$ = 0.0115 gms. HCOOH.

Average Analysis

The two determinations should give substantially the same result. If the first is much higher, adulteration with a mineral acid is quite probable. Formic acid is usually sold in 60% strength.

No. 4 ACID—HYDROCHLORIC

(Muriatic Acid)

HCl

Specific Gravity

Determine by hydrometer at 15° C.

Acidity

Weight into a glass stoppered weighing bottle 0.2 to 0.4 gms. of the sample. Wash into a beaker, dilute with distilled water and titrate with N/10 NaOH, using Methyl Red as an indicator. Calculate as HCl. 1 cc. N/10 NaOH = 0.003647 gms. HCl.

Residue

Pipette 100 cc. of the sample into a weighed platinum evaporating dish. Evaporate to dryness on the steam bath, then heat for at least one hour in the oven at 100° C., cool in a desiccator and weigh.

Sulfates

Test qualitatively for sulfates. If the presence of more than a trace is indicated, determine quantitatively as described under sodium sulfate (No. 72).

Iron

Test qualitatively for iron. If the presence of more than a trace is indicated, determine quantitatively as follows: Transfer 1 cc. of the sample to a 50 cc. Nessler tube with a little water. Add N/2 $KMnO_4$ drop by drop until a pink color persists for 5 minutes. Then add 5 cc. of a normal potassium sulfocyanate (KSCN) solution and make up to the mark. Make a standard for comparison by adding 45 cc. distilled water and 5cc. N/1 KSCN

to another tube. Finally add small measured amounts of standard iron solution until the color of the standard matches that of the sample. Calculate as Fe.

1 cc. standard iron solution = 0.0001 gm. Fe.

Average Analysis

Specific Gravity	1.16
Acidity as HCl	30.0%
Residue	Not over 0.2
Sulfates	Not over 0.2

No. 5 ACID—LACTIC
$CH_3.CHOH.COOH$

Specific Gravity

Determine by hydrometer at 15° C.

Acidity

Pipette out 5 cc. of the sample and dilute to exactly 250 cc. with water. Use 25 cc. aliquots and titrate with N/10 NaOH, using Phenolphthalein as an indicator. Calculate as $C_3H_6O_3$.

1 cc. N/10 NaOH = 0.009 gms. $C_3H_6O_3$.

Average Analysis

Specific Gravity	1.18
Acidity as $C_3H_6O_3$	50%

No. 6 ACID—NITRIC
HNO_3

Specific Gravity

Determine by hydrometer at 15° C.

Acidity

Weigh into a glass stoppered weighing bottle 0.2 to 0.4 gms. of the sample. Wash into a beaker, dilute with distilled water and titrate with N/10 NaOH, using Methyl Red as an indicator. Calculate as HNO_3.

1 cc. N/10 NaOH = 0.0063 gms. HNO_3.

Residue

Pipette 100 cc. of the sample into a weighed platinum evaporating dish. Evaporate to dryness on the steam bath, then heat for at least one hour in oven at 100° C., cool in a desiccator and weigh.

Qualitative Tests

If the residue is appreciable, make qualitative tests for lead, copper, iron, chlorides and sulfates.

Average Analysis of Nitric Acid (42° Be)

Specific Gravity	1.406
Acidity, as HNO_3	66.8%
Residue	Not over 0.2%
Color	Clear and colorless

No. 7 ACID—OXALIC
$H_2C_2O_4 2H_2O$

Acidity

Weigh out from 0.5 to .0 gm. of the sample and dissolve in water in a beaker. Acidify with dilute sulfuric acid (about 15 cc.), heat nearly to boiling, then titrate with N/10 potassium permanganate. Calculate as $H_2C_2O_4$.

1 cc. N/10 $KMnO_4$ = 0.0045 gms. $H_2C_2O_4$.

Average Analysis

Acidity as $H_2C_2O_4$	70%

No. 8 ACID—SULFURIC
(Oil of Vitriol)
H_2SO_4

Specific Gravity

Determine by hydrometer at 15° C.

Acidity

Weigh into a glass stoppered weighing bottle 0.15 to 0.25 gms. of the sample. Wash into a beaker, dilute with distilled water and titrate with N/10 NaOH using Methyl Red as an indicator. Calculate as H_2SO_4.

1 cc. N/10 NaOH = 0.0049 gms. H_2SO_4.

Residue

Weigh about 10 gms. into a platinum evaporating dish and evaporate to dryness over a low flame (*in the hood*), then ignite at low red heat for several minutes, cool and weigh.

Qualitative Tests

Iron: Take 10 cc. of the sample and dilute to 100 cc. with water, then make distinctly alkaline with dilute ammonia and note whether brownish precipitate appears on standing.

Lead: Mix a portion of the acid with 5 times its volume of strong alcohol and note whether turbidity appears.

Average Analysis

Specific Gravity	1.827
Acidity, as H_2SO_4	91.5%
Residue	Not over 0.05%
Iron and Lead	Not more than a trace
Color	Should be clear and colorless

No. 9 ACID—SULFUROUS
H_2SO_3

Specific Gravity

Determine by hydrometer at 15° C.

Acidity

Weigh into a glass stoppered weighing bottle 0.2 to 0.4 gms. of the sample. Wash into a beaker, dilute with distilled water and titrate with N/10 NaOH, using Phenolphthalein as an indicator. Calculate as H_2SO_3.

1 cc. N/10 NaOH = 0.0041 gms. H_2SO_3.

Sulfur Dioxide

Weigh out about 5 gms. of the sample and dilute to one liter. Pipette out 10 cc. of this solution, add about 100 cc. of water and 5 cc. of starch solution and titrate with N/10 iodine until a blue color appears. Calculate as SO_2.

1 cc. N/10 iodine = 0.0032 gms. SO_2.

Residue

Pipette 100 gms. of the sample into a weighted platinum evaporating dish and evaporate slowly on an asbestos

board heated by a free flame until all fumes have ceased. Cool and weigh.

Average Analysis

Specific Gravity	1.035
Acidity as H_2SO_3	3.5%
Reducing Power as SO_2	3.9%
Residue	Not over 2.2%

No. 10 ACID—TANNIC

Tannin Estimation

Total oxidizable matters:

2 gms. of the sample are dissolved in water and the solution diluted to 500 cc. 10 cc. of this solution and 25 cc. of indigotin (0.1%) are diluted with 500 cc. of water and titrated with N/50 potassium permanganate until a pure yellow color is obtained. 25 cc. of indigotin solution alone are similarly titrated, and the difference calculated in terms of tannin.

Oxidizable matters other than Tannin:

20 cc. of the tannin solution are well shaken in a glass stoppered bottle with 40 cc. of gelatine solution and 40 cc. of acid salt solution, then filtered. 25 cc. of the filtrate and 25 cc. of indigotin are diluted with 500 cc. water and titrated as above. Then 25 cc. of indigotin, mixed with 10 cc. of gelatine and 10 cc. of acid salt solution are similarly titrated, and the difference calculated as above.

1 cc. N/50 $KMnO_4$ = 0.0008314 grams tannin.

Actual tannin:

Subtraction of the second result from the first gives the actual tannin.

Ash

Ignite about 1 gm. in a platinum crucible.

Moisture

Weigh about 1 gm. into glass stoppered weighing bottle and dry at 110° C. for 3 hours.

Average Analysis for U. S. P. Grade

Moisture	10%
Ash	Under 0.30%
Tannins	97%
Non-Tannins	9.5%

No. 11 ACID—TARTARIC
$COOH(CHOH)_2COOH$

Acidity

Weigh about 0.2 gms. of the sample into a beaker, dissolve in water and titrate with N/10 NaOH using Phenolphthalein as an indicator. Calculate as $C_4H_6O_6$.

1 cc. N/10 NaOH = 0.0075 gms. $C_4H_6O_6$.

Ash

Ignite about 1 gm. of the sample in a platinum crucible.

Average Analysis

Acidity as $C_4H_6O_6$	99.97%
Ash	Not over 0.03%

No. 12 ALCOHOL
C_2H_5OH

Specific Gravity

Determine by hydrometer at 15° C. The percentage of alcohol by volume and by weight may be calculated from the specific gravity. Consult the tables in Van Nostrand's Chemical Annual or a similar handbook.

NOTE:— Make only this test in the case of denatured alcohol. The remaining determinations apply to grain alcohol.

Acidity

Take 100 cc. of the sample, add a little Phenolphthalein and titrate with N/10 NaOH until the pink tinge persists. Calculate as acetic acid.

1 cc. N/10 NaOH = 0.006 gms. $HC_2H_3O_2$.

Residue

Pipette 100 cc. of the sample into a weighed platinum evaporating dish and evaporate slowly to dryness. Cool and weigh.

Foreign Organic Impurities

When 1 cc. of N/10 $KMnO_4$ is added to 100 cc. of grain alcohol, the pink color should remain at least 20 minutes.

Aldehyde

10 cc. of the sample are mixed in a test tube with 1 cc. of water and 5 drops of silver nitrate solution. The solution must not become turbid nor colored on heating.

Average Analysis

	Denatured	*Grain*
Specific Gravity at 15° C.	0.817	0.819
Alcohol by volume		94.10%
Alcohol by weight		91.10%
Acidity as $HC_2H_3O_2$		Not over 0.02%
Residue		Not over 0.005%

No. 13 ALUMINUM CHLORIDE
$AlCl_3$

Specific Gravity

Determine by hydrometer at 15° C.

Alumina

Dissolve 5 grams in 500 cc. and titrate 25 cc. aliquots with N/10 NaOH using Phenolphthalein as indicator. Calculate as $AlCl_3$.

1 cc. N/10 NaOH = 0.00445 gms. $AlCl_3$.

Average Analysis

Specific gravity at 15°C.	32° Be
Aluminum Chloride ($AlCl_3$)	27.5%

No. 14 ALUMINUM SULFATE
(Sulfate of Alumina)
$Al_2(SO_4)_3.18H_2O$

Alumnia

Weigh out 1 gm. of the sample and dissolve in about 200 cc. of boiling water. (Note: if there is insoluble

(Continued on page 485)

AMERICAN DYESTUFF REPORTER

Devoted to the use and application of dyestuffs and the mechanical equipment incidental thereto

LOUIS A. OLNEY
Professor of Chemistry and Dyeing, Lowell Textile Institute
Directing Editor

NORMAN A. JOHNSON
Managing Editor

MYRON DREW REESER
Advertising Manager

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ABOUT DESIGN PROTECTION

ONE of the foremost organizations in the textile industries—The United Piece Dye Works—has taken a courageous step, and in taking it, may have paved the way for the passage of legislation that these industries have been trying vainly to enact for years. The United, for its stand alone, should be praised because of its foresight and exonerated of any charges that might be brought by ill advised or uninformed visionaries. If its new policy does throw so strong a light on the pitifully inadequate conditions relating to fabric patterns as to awaken Congress to the realization that design protection legislation must be enacted immediately, then the United will have established itself as a benefactor of the designing, printing and fabric trades.

In self defense, the United has been obliged to pass a ruling enabling its organization to accept, for printing and engraving, designs that have not been registered by the Design Protection Association. The United has always been recognized as a pioneer in practices intended to benefit the industry and as an upholder of the highest standards of ethics. Last December, when engravers and printers agreed to refuse work on designs that could not be proved to be original, the United was enthusiastic in taking a leading role in thus limiting its business opportunities. Only a few months have been needed, however, to prove to the United that this attempt was not the practical, effective means of eliminating design piracy. The United, therefore, refused to continue to support the policy, and in refusing showed that it had the intelligence and courage to acknowledge that it had been wrong. That this is the general attitude of leaders of the affected trades is indicated by their approval of the United's decision to change its policy.

In connection with the foregoing we call the attention of our readers to the article "Design Copyright for Textiles Only" on page 477 of this issue. Although written before the move was made by the United Piece Dye Works it offers some encouragement to textile interests.

FURTHER PROTECTION FOR THE CONSUMER

THE movement which is now on foot to provide for legislation requiring the labeling of textile goods to show the materials of which they are composed is one which should receive the cooperation of all legitimate branches of the industry. Such a movement is in direct accord with the fast advancing demand for quality fabrics rather than cheap fabrics sold wholly on a price basis. Such a labeling system when properly worked out will act as a guarantee of quality to the buyer. This movement is said to have the support of the Throwsters Research Institute as it is thought that it will put the silk industry on a sounder basis as well as giving the public protection by giving them information as to whether they were getting silk or an imitation of silk. The same thing would be true of other types of textile fabrics. The reaction of the silk industry to the movement may possibly be expressed by the following excerpts clipped from the *Paterson Call.*

"Synthetic products, resembling silk, have had a tremendous growth, being used largely in the making of all kinds of underwear, women's dresses, neckties, etc., sometimes alone and frequently in combination with silk, they being sold presumably as silk, although honest merchants are ready to tell inquirers whether they are silk, silk in combination with other textile fibers, or a synthetic product entirely. Many people however, make no such inquiries and take it for granted that because the goods they are buying look like silk they are silk.

"Such legislation, while undoubtedly a benefit to the silk industry as a whole, would not be injurious to the synthetic industry, outside of the fact that such synthetic products would have to stand on their own bottom instead of masquerading in the minds of purchasers as silk. The synthetic industry has made such rapid strides in improving the quality and beauty of its product, as well as its durability, that there is no need for it to masquerade as something it looks like but is not."

Other forces working to this end are the energetic promotions of the Department of Commerce along this line and the efforts of the National Quality Maintenance League to focus the attention of the purchasing public upon the quality of a fabric rather than on its price.

Further than labeling goods it is necessary to educate the public to read them in order to receive the full protection afforded by such means. The labeling system has long been employed in the food and drug industry to protect the public but it is only within the last several months that the U. S. Food and Drug Administration has carried on a "Read-the-Label" campaign to teach the public the value of labeling goods.

1932 Fall Color Card

The regular edition of the 1932 Fall Season Silk Card has just been released to members, it was announced recently by the Textile Color Card Association.

Browns, of first importance in the fall color mode, are prominently featured. Brandy, Manila Brown and Marron, the latter a tête de nègre tone, represent outstanding types, while Rhum Brown and Mahogany interpret the smart reddish note in browns. Malaga Brown and Catawba Brown as their names suggest, reflect the new wine cast.

Vintage tones, including Médoc and Bordeaux Red, are also stressed as a high fashion development. Rouge Laque and Cocktail Red, gay, vivid hues of orange cast, are especially adapted to sportswear.

Blues, which are expected to maintain their strong position for early fall, include clear intense tones, such as Polo Blue and Jockey Blue and soft violet-tinted shades like Bleu Pervenche and Brioni Blue. Tailleur Blue and Deepblu are new renditions of navy. Less prominence is accorded green in the fall color picture. Talisman Green, of slight yellowish cast, is a clear bright hue, while Rustic Green is a darker basic tone.

In the beige family neutral sandy shades, such as String and Mastic Beige, are emphasized. Dusty pinks, deep mustardy yellows, coffee-tinted beige, grège and a clear, light grey are cited as new accent tones.

Special groups highlighted on the card include chalky off-whites and crayon pastels for evening wear and gay vivid hues for contrast themes.

Basic Colors for Fall

Black is expected to be the most popular color for coats and suits this fall, according to the Color Coordination Committee of the National Retail Dry Goods Association, which has announced its shades for the Fall Color Card.

Black, it is anticipated, will account for over 50 per cent of the business in the coming season, since it will be popular both as high style and for popular priced garments.

Next in popularity, the committee predicts, will be a neutral tone of Brown, with Red Brown coming next in importance from the viewpoint of volume sales.

Wine Red and Port Wine are also popular fall shades, selected for the Color Card, and deep Woodland Green and a Military Blue are also colors which will be of importance during the coming season.

These same shades are also shown as the basic wool dress colors, and the silk shades chosen as basic for fall buying are similar to those chosen for coats and suits. In addition to these harmonious shades for costume ensembles, the committee has chosen additional brighter sportswear colors that are considered of volume importance.

They include: Rum Brown, Natural Beige, Rust Red, Cruuson Red, Irish Green, Pistache Green and Chinese Blue.

Millinery Colors

The Color Conference Committee of the Eastern Millinery Association in cooperation with the Textile Color Card Association, has chosen nineteen colors for early fall wear, according to an announcement just made by the color organization.

These new shades, which will be promoted by all factors of millinery and allied lines, coordinate closely with the basic shades for early fall sponsored by the textile and related industries.

Siamese Dye Imports Steady

Preliminary indications from Siam show that, although there has been a drastic decline in imports of most commodities, incoming shipments of aniline dyes will remain at practically the same level. Bangkok is the chief port of entry and imports through this port for the fiscal year ending March 31, 1932, totaled 103,095 kilos worth 230,776 ticals, compared to total imports for all Siam of 110,447 kilos, worth 253,268 ticals in 1930-31. It is believed that Germany continued to receive practically all of the dye business, according to Assistant Trade Commissioner Joe D. Walstrom, Bangkok.

1932 Fall Correlation Chart

The 1932 Fall Correlation Chart, which will serve primarily as a guide to retailers in coordinating the new costume and accessory shades, has just been released to members, it was announced recently by the Textile Color Card Association.

The principal basic ensemble and millinery colors are listed in the chart according to their cast and for each classification are indicated the proper shades for shoes, bags, hosiery, gloves and jewelry. For example, a costume in the fashionable Rhum Brown range may be correctly complemented by shoes, bag and gloves of Madeira Brown, Rhumtone hosiery and jewelry in the contrasting Mustard Gold hue. Porto, the new vintage red leather tone, is mentioned as the correct shade for shoes and bag to accompany the Bordeaux Red costume, with which Praline Pink jewelry is a smart accent note.

New themes in color harmonies and contrasts are also indicated in the chart, which is to be used in conjunction with the 1932 Fall Silk, Woolen, Shoe and Leather and hosiery cards already released by the Textile Color Card Association.

This color guide supplements the special charts issued earlier by the Association to each membership group, in which basic colors were shown in silk and woolen swatches, together with their coordinating hosiery, shoe and bag shades.

Analytical Methods for a Textile Laboratory—Continued

(Continued from page 482)

matter, this should be filtered off and weighed). Then add 25 cc. of ammonium chloride solution and a very slight excess of dilute ammonia. Keep the solution hot for about ½ hour, then filter and wash with hot water. The precipitate is then dried, ignited in a blast lamp, and weighed as Al_2O_3.

Basicity or Acidity

Dissolve 25 gms. of the sample in boiling water and dilute to 500 cc. Place exactly 68 cc. of this solution (equivalent to 3.40 gms. of the sample) in a 4-inch casserole, add about 35 cc. of distilled water and heat to boiling. Add exactly 10 cc. of N/2 H_2SO_4, cool to room temperature and add 18 to 20 cc. of potassium fluoride solution and a few drops of Phenolphthalein. Titrate with N/2 KOH (do not use NaOH), adding it drop by drop until a slight pink, persisting for 1 minute, is obtained.

Free Al_2O_3—When the back titration with KOH is less than the amount of H_2SO_4 added, the difference is calculated in terms of Al_2O_3.

% of free Al_2O_3 = (cc. of H_2SO_4 — cc. of KOH) x 0.25.

Free H_2SO_4 — When the back titration with KOH is greater than the amount of H_2SO_4 added, the difference is calculated in terms of H_2SO_4.

% of free H_2SO_4 = (cc. of KOH — cc. of H_2SO_4) x 0.72.

Neutrality — When the back titration with KOH is equal to the amount of H_2SO_4 added, the sample is neutral.

Iron

Test for this as usual with potassium ferrocyanide.

Average Analysis

Alumina as Al_2O_3..... 17%

No. 15 AMMONIUM HYDROXIDE (Ammonia) NH_4OH

Specific Gravity

Pour the sample into the gravity jar and take the gravity immediately. Then note the temperature and make corrections to 15°C. Consult the tables in Van Nostrand's Chemical Annual or a similar handbook.

Alkalinity

Measure from a burette 10 cc. of the sample into a weighed weighing bottle, stopper tightly and weigh again. Measure with the same burette another 10 cc. of the sample into a 500 cc. volumetric flask partly filled with distilled water and then dilute to 500 cc. Use 10 cc. aliquots, add a slight excess of N/10 HCl, and titrate the excess with N/10 NaOH, using Methyl Red as an indicator. Calculate as NH_3.

1 cc. N/10 HCl = 0.0017 gms. NH_3

Qualitative Tests

Test as usual for chlorides and sulfates.

Average Analysis

Specific Gravity at 15°C.....0.90
Color .....Clear and Colorless
Alkalinity as NH_3.....27.8%

No. 16 AMMONIUM ALUMINUM SULFATE (Ammonia Alum-Ground) $(NH_4)_2SO_4.Al_2(SO_4)_3.24H_2O$

Alumina

Determine as in Aluminum Sulfate (No. 14).

Free Acid

Weigh out about 5 gms. of the sample, add 100 cc. of neutral 95% alcohol, and warm for 15 minutes with constant stirring. Filter, wash with a little more alcohol, and titrate the filtrate with N/10 NaOH using Phenolphthalein as an indicator. Calculate as sulfuric acid.

1 cc. N/10 NaOH = 0.0049 gms. H_2SO_4.

Average Analysis

Alumina as Al_2O_3..... 11.5%
Acid as H_2SO_4..... 2.0%

No. 17 AMMONIUM CHLORIDE (Sal Ammoniac) NH_4Cl

Strength Determination

Dissolve about 1 gram of the sample in a little water or take an equivalent amount if the substance is a liquid. Add 25 cc. of a neutral 20% formaldehyde solution. Warm the mixture to 60°C. and titrate at this temperature with N/2 NaOH using Phenolphthalein as an indicator. Calculate as NH_4Cl.

1 cc. N/2 NaOH = 0.02675 gms. NH_4Cl.

Average Analysis

Ammonium Chloride as NH_4Cl...98 to 99%

No. 18 AMMONIUM SULFATE $(NH_4)_2SO_4$

Strength Determination

Dissolve about 1 gram of the sample in a little water

or take an equivalent amount if the substance is a liquid. Add 25 cc. of a neutral 20% formaldehyde solution. Warm the mixture to 60°C. and titrate at this temperature with N/2 NaOH using Phenolphthalein as an indicator. Calculate as $(NH_4)_2SO_4$.

1 cc N/10 NaOH = 0.033 gms. $(NH_4)_2SO_4$.

Average Analysis

Ammonium Sulfate as $(NH_4)_2SO_4$.....98 to 99%

No. 19 ANTIMONYL POTASSIUM TARTRATE

(Tartar Emetic)

$K(SbO)C_4H_4O_6.\frac{1}{2}\ H_2O$

Antimony

Grind the crystals to a powder and weigh about 0.4 gms. into a beaker. Dissolve in water, warming if necessary, and then to the cold solution add 25 cc. of a cold saturated solution of sodium bicarbonate and titrate with N/10 iodine using starch as an indicator. Calculate as Sb.

1 cc. N/10 iodine = 0.006 gms. Sb

Average Analysis

Antimony as Sb..... 35.8%

No. 20 BEESWAX

Melting Point

Draw out a small piece of glass tubing to a fine point. Melt a little of the wax at a low temperature and draw it up into this tube, then let stand over night. Attach tube to end of thermometer and immerse in a beaker of water. Place this beaker in a pan of water and heat very slowly. Note the exact temperature at which the wax melts.

Saponification Value

5 gms. of the sample are boiled under a reflux condenser for at least 1¼ hours with 25 cc. of N/2 alcoholic potash. The excess of alkali is then titrated with N/2 HCl using Phenolphthalein as an indicator. Calculate mgms. of KOH per gram of wax.

1 cc. N/2 KOH = 28.05 mgms. KOH

Acid Value

8 gms. of the sample are heated in a flask with 50 cc. of neutral absolute alcohol until perfectly melted, then titrated with N/2 alcoholic potash, using Phenolphthalein as an indicator. Calculate as above.

Ester Value

This is the difference between the acid value and the saponification value.

Ester Ratio

This is the ratio of the ester value to the acid value.

Average Analysis

Melting Point	65°C.
Saponification Value	90 to 96
Acid Vlaue	18 to 22
Ester Value	70 to 80
Ester Ratio	3.2 to 4.2

No. 21 BETA NAPHTHOL

$C_{10}H_7OH$

Melting Point

Place a few grains of the sample in a capillary tube sealed at one end. Attach this tube to the bulb of a thermometer and immerse partly in a bath of concentrated sulfuric acid. Heat this bath slowly and note carefully the temperature at which the substance melts.

Ash

Ignite one gram in a platinum crucible.

Average Analysis

Melting Point	121-122°C.
Ash	None

No. 22 CALCIUM CHLORIDE

$CaCl_2.H_2O$

Lime

Weigh about 1 gm. of the sample into a beaker and dissolve in 150 cc. water. Acidify with 15 cc. dilute HCl, heat nearly to boiling, and add 50 cc. of ammonium oxalate solution. Then while still hot, add dilute ammonia with constant stirring until the solution is alkaline. Continue the heating for several minutes, then let stand over night. Filter, wash with hot water, ignite, and weigh the resulting calcium oxide. Calculate as CaO or $CaCl_2$. Multiply CaO by the factor 1.9793 to obtain $CaCl_2$.

Average Analysis

Lime as CaO	40%
Calcium Chloride	79%

No. 23 CALCIUM HYPOCHLORITE

(Bleaching Powder) (Chloride of Lime)

$Ca(OCl)_2$

Available Chlorine

10 gms. of the sample are weighed out into a mortar and rubbed to a thin cream with water. After allowing the coarser particles to subside, the turbid liquid is poured into a liter flask. The process is repeated with more water until everything is transferred to the flask which is then filled to the mark and well shaken. 50 cc. of the turbid solution is immediately pipetted out, 50 cc. of N/10 sodium arsenite solution added, and the excess then titrated with N/10 iodine solution. Calculate as Cl.

1 cc. N/10 solium arsenite = 0.003546 gms. Cl.

Average Analysis

Total available chlorine..... 35%

No. 24 CASEIN

Ash

Weigh from 1.0 to 2.0 gms. of the sample into a plati-

num crucible and ignite to a constant weight at low red heat.

Nitrogen

Weigh from 0.2 to 0.3 gms. of the sample into a Kjeldahl flask and add 20 cc. of conc. sulfuric acid and 15 gms. potassium sulfate. Heat with a low flame under the hood until the liquid is clear and colorless. Then dilute with 150 to 200 cc. of water, add an excess (about 100 cc.) of caustic soda solution (30°Be) and a small quantity of talc (or granular zinc) to prevent bumping. Distill the larger portion of the liquid from the flask into a beaker containing 50 cc. N/10 HCl and 100 to 300 cc. water, with the delivery tube below the surface of the solution. Then titrate the excess of acid in the beaker with N/10 NaOH using Methyl Red as an indicator. Calculate as N.

1 cc. N/10 HCl = 0.0014 gms. N.

Note: A blank should be run on the reagents in the same manner as described above.

Moisture

Weigh about 1 gm. of the sample into a weighing bottle and dry in the oven at 50°C. to a constant weight.

Average Analysis

Ash	3.5%
Nitrogen	14.7%
Moisture	5.5%

No. 25 COAL

Determination of Heat Units

This analysis is conducted in the bomb calorimeter according to the directions given for operating the same. After making the necessary corrections, the heat units are calculated first in calories per gram, then in B.T.U. per pound.

Calories per gm. x 1.8 = B.T.U. per lb.

Moisture

Dry a 1 gm. sample in an oven for 1 hour at 110°C., cool in a desiccator and weigh.

Volatile Matter

Weigh about 1 gm. of the sample into a platinum crucible, place a cover on the same, and heat for exactly seven minutes, using a gas burner of size sufficient so that the flame entirely surrounds the crucible. Cool in a desiccator and weigh.

Ash

Weigh about 1 gm. of the sample into a porcelain crucible and ignite to constant weight, heating very slowly and carefully at first until the volatile matter is driven off.

Fixed Carbon

This is determined indirectly by adding together the percentage of moisture, volatile matter, and ash, and subtracting the sum from 100.

Sulfur

After the heat test in the bomb calorimeter, wash the remaining contents carefully into a glass beaker. Make slightly alkaline with dilute ammonia, heat to boiling, filter, and wash. Acidify the filtrate with dilute hydrochloric acid, add a few drops of bromine water, and boil until the yellow color of the bromine is driven off. Then precipitate with barium chloride as usual, weigh barium sulfate and calculate as sulfur. Multiply $BaSO_4$ by the factor 0.1373 to obtain S.

No. 26 CUPRIC ACETATE
(Acetate of Copper)
$Cu(C_2H_3O_2)_2.H_2O$

Copper

Dissolve about 1 gm. of the sample in 20 cc. of water, add 10 cc. of dilute ammonia, then neutralize with dilute acetic acid. Add a slight excess of acid, then about 5 gms. of potassium iodide and titrate with N/10 sodium thiosulfate until the iodine color disappears, using starch near the end of the titration. Calculate as Cu or as $Cu(C_2H_3O_2)_2$.

1 cc. N/10 $Na_2S_2O_3$ = 0.00636 gms. Cu

1 cc. N/10 $Na_2S_2O_3$ = 0.01816 gms. $Cu(C_2H_3O_2)_2$

Qualitative Test

Test as usual for sulfates. If more than a trace is present, determine quantitatively.

Average Analysis

Total Copper as Cu	31.5%
Copper Acetate as $Cu(C_2H_3O_2)_2$	90.0%

No. 27 CUPRIC SULFATE
(Blue Vitriol)
$CuSO_4.5H_2O$

Copper

Dissolve about 1 gm. of the sample in 20 cc. of water, add 10 cc. of dilute ammonia, then neutralize with dilute acetic acid. Add a slight excess of acid, then about 5 gms. of potassium iodide and titrate with N/10 sodium thiosulfate until the iodine color disappears, using starch near the end of the titration. Calculate as Cu or as $CuSO_4$.

1 cc. N/10 $Na_2S_2O_3$ = 0.00636 gms. Cu

1 cc. N/10 $Na_2S_2O_3$ = 0.01596 gms. $CuSO_4$

Iron

Test for this as usual.

Average Analysis

Copper as Cu	24.85%
Copper Sulfate as $CuSO_4$	62.36%

No. 28 DEXTRINE
(British Gum)

Strength Determination

Weigh two grams of the sample, dissolve in water, and make up to exactly 500 cc. Take 100 cc. aliquots and boil three hours with 10 cc. HCl (Sp. Gr. 1.125), using a return condenser. Cool and nearly neutralize with 10% NaOH. Determine the dextrine with Fehling's

solution, using 50 cc. aliquots as follows:—add the dextrine solution to a mixture of 25 cc. each of Fehling's solutions I and II, heat to a boil within four minutes and boil exactly two minutes. Filter on a weighed Gooch crucible with asbestos mat, dry, and weigh as Cu_2O. Calculate to dextrose by consulting the tables in Molinari's Industrial Organic Chemistry, 2nd Edition, 1923, Page 582. Multiply dextrose by the factor 0.9 to obtain dextrine.

Moisture

Weigh five grams and dissolve in 200 cc. water, pipette 50 cc. portions into moisture dishes, dry and weigh. Calculate the moisture.

Insoluble Matter

Pipette two 50 cc. portions of this solution into centrifuge tubes and whirl for twenty minutes at fairly high speed to settle out the insoluble matter. Pipette 25 cc. aliquots into moisture dishes, being careful not to stir up the sediment. Evaporate to dryness, dry in the oven, cool and weigh. Calculate the insoluble matter by difference.

Ash

Burn about one gram to constant weight in a platinum crucible and calculate the ash.

Average Analysis of Corn Dextrine

Dextrine	87 to 94%
Ash	.10%
Moisture	7.00%
Insoluble Matter	6.50%

No. 29 DYESTUFFS

Estimation of Na_2SO_4

Weigh 0.5 to 1.0 gm. of the dye into a porcelain crucible. Then burn until all the material has become thoroughly charred. It is not necessary to entirely burn off the carbon. Add a small amount of distilled water to dissolve the inorganic salts, then wash everything into a beaker. Heat nearly to boiling, filter to remove the carbon particles, then wash with hot water. Determine the sulfate present in the solution in the usual manner by precipitation with $BaCl_2$. (See method under Sodium Sulfate, No. 72). Weigh as $BaSO_4$ and multiply by the factor 0.608 to obtain Na_2SO_4.

Estimation of NaCl

Weigh 0.2 to 0.4 gms. of the dye into a porcelain crucible. Burn as above, but at a lower heat so as to prevent any volatilization of the NaCl. Dissolve the inorganic salts in water and wash into a beaker. Dilute with 50 to 75 cc. of water, add a few drops of potassium chromate solution and titrate with N/10 $AgNO_3$ until the solution assumes a faint red tinge. Calculate as NaCl.

1 cc N/10 $AgNO_3$ = 0.005845 gms. NaCl.

No. 30 FERRIC CHLORIDE
(Chloride of Iron)
$FeCl_3$

Specific Gravity

Determine with the hydrometer at 15°C.

Total Iron

Weigh out about 2 gms. of the sample, add 200 cc. of water and 10 cc. of dilute nitric acid, then boil for several minutes. Add a slight excess of dilute ammonia, heat to boiling, then filter and wash. The precipitate is placed in a platinum crucible and ignited to constant weight. Weigh as Fe_2O_3. Multiply by the factor 0.6994 to obtain Fe.

Average Analysis

Specific Gravity	1.401
Total Iron as Fe	11.7%

No. 31 FERRIC SULFATE
(Nitrate of Iron) ....
$Fe_2(SO_4)_3$

Specific Gravity

Determine with the hydrometer at 15°C.

Ferrous Iron

Weigh out about 10 gms. of the sample, dilute with 100 cc. of water, add 25 cc. of dilute sulfuric acid and titrate with N/10 potassium permanganate. Calculate as FeO.

1 cc. N/10 $KMnO_4$ = 0.007184 gms. FeO

Total Iron

Weigh out about 2 gms. of the sample, add 200 cc. of water and 10 cc. of dilute nitric acid, then boil for several minutes. Add a slight excess of ammonia, heat to boiling, then filter and wash. The precipitate is placed in a platinum crucible and ignited to constant weight. Weigh as Fe_2O_3. Multiply by the factor 0.6994 to obtain Fe.

Acid to Base

Weigh out about 2.5 to 3 gms. of the sample, add 100 cc. of water and exactly 25 cc. of normal sodium carbonate. Heat to boiling, filter and wash with hot water. Titrate the excess of sodium carbonate in the filtrate with N/1 or N/2 HCl using Methyl Orange as an indicator. Calculate as Sulfur Trioxide. 1 cc. N/1 Na_2CO_3 = 0.04003 gms. SO_3. Also calculate the amount of Sulfur Trioxide necessary to form normal sulfates with the iron present.

Hydrochloric Acid

Test for this in the usual manner.

Average Analysis

Specific Gravity	1.492
Total Iron as Fe	11.92%
Ferrous Oxide as FeO	10.90%
Ferric Oxide as Fe_2O_3	16.04%
SO_3 Found	22.08%
SO_3 Theoretical	25.11%

No. 32 FERROUS ACETATE
(Black Iron Liquor)
$Fe(C_2H_3O_2)_2$

Specific Gravity

Determine with a hydrometer at 15°C.

Total Iron

Weigh about 4 grams into a platinum evaporating dish, evaporate to dryness on the steam bath, and ignite until all the organic matter is burned off. Then add dilute hydrochloric acid and heat on the steam bath until the iron is dissolved. Remove from evaporating dish, add 10 cc. of dilute nitric acid, bring to a boil to change all ferrous iron to ferric, and then precipitate with dilute ammonia. Continue boiling for a minute and filter, dry, ignite and weigh as Fe_2O_3. Multiply by the factor 0.6994 to obtain Fe. Save the filtrate, acidify it with dilute hydrochloric acid, and add barium chloride to detect the presence of any sulfates. If there is more than a trace, determine quantitatively, otherwise report as a trace.

Average Analysis

Specific Gravity	1.10-1.12
Total Iron	3.50%-5.07%
Sulfates	None

No. 33 FERROUS SULFATE
(Copperas)
$FeSO_4.7H_2O$

Ferrous Iron

Weigh out 1 gm. of the sample, add 100 cc. of water and 50 cc. of dilute sulfuric acid, then titrate with N/10 potassium permanganate. Calculate as Fe or as $FeSO_4.7H_2O$.

1 cc. N/10 $KMnO_4$ = 0.005584 gms. Fe
1 cc. N/10 $KMnO_4$ = 0.0278 gms. $FeSO_4.7H_2O$

Ferric Iron

Test for this with potassium ferrocyanide.

Average Analysis

Iron as Fe	19.84%
Ferrous Sulfate as $FeSO_4.7H_2O$	98.78%
Ferric Iron	Not more than a trace

No. 34 FORMALDEHYDE
(HCHO)

Specific Gravity

Determine with hydrometer at 15°C.

Quantitative Estimation

Ten grams of the sample are diluted with water to 500 cc. and 5 cc. aliquots used. Forty cc. of N/10 Iodine solution are added and sodium hydroxide solution (30° Be) a drop at a time until the color of the solution is light yellow. Then let stand exactly ten minutes. Acidify with dilute hydrochloric acid and titrate the unused iodine with N/10 sodium thiosulfate. Calculate as HCHO.

1 cc. N/10 Iodine solution = 0.0015 gms. HCHO.

Free Acid

Weigh about 10 gms. of the sample into a stoppered flask, dilute and titrate with N/10 NaOH, using Phenolphthalein as an indicator. Calculate as HCOOH.

1 cc. N/10 NaOH = 0.0046 gms. HCOOH

Average Analysis

Specific Gravity	1.085
Strength as HCHO	37.5%
Acidity as HCOOH	0.08%

No. 35 GALL EXTRACT

Tannin Estimation

Total Oxidizable Matter.

5 gms. of the sample are diluted to 500 cc. 10 cc. of this solution and 25 cc. of indigotin (0.1%) are diluted with 500 cc. of water and titrated with N/50 potassium permanganate until a pure yellow color is obtained. 25 cc. of indigotin solution alone are similarly titrated, and the difference calculated in terms of tannin.

Oxidizable Matter other than tannin.

20 cc. of the tannin solution are well shaken in a glass stoppered bottle with 40 cc. of gelatine solution and 40 cc. of acid salt solution, then filtered. 25 cc. of the filtrate and 25 cc. of indigotin are diluted with 500 cc. water and titrated as above. Then 25 cc. of indigotin, mixed with 10 cc. of gelatine and 10 cc. of acid salt solution are similarly titrated, and the difference calculated as above.

1 cc. N/50 $KMnO_4$ = 0.0008314 gms. tannin

Actual tannin

Subtraction of the second result from the first gives the actual tannin.

Average Analysis

Total Oxidizable Matter	45%
Non-tannin	10%
Tannin	35%

No. 36. GELATINE

Nitrogen

Weigh 0.4 grams of the sample into a Kjeldahl flask and add 20 cc. of conc. sulfuric acid and 15 grams of potassium sulfate. Heat with low flame under the hood until the liquid is clear and colorless. Then dilute with 150 to 200 cc. of water, add an excess (about 100 cc.) of caustic soda solution (30° Be) and a small quantity of talc (or granular zinc) to prevent bumping. Distill the larger portion of the liquid from the flask into a beaker containing 50 cc. N/10 HCl and 100 to 300 cc. of water, with the delivery tube below the surface of the solution. Then titrate the excess of HCl in the beaker with N/10 NaOH using Methyl Red as an indicator. Calculate as N.

1 cc. N/10 NaOH = 0.0014 gms. N.

Note: A blank should be run on the reagents in the same manner as described above.

Moisture

Weigh about 1 gram of the sample into a glass stoppered weighing bottle and dry at 110°C. for three hours.

Ash

Weigh about 1 gram of the sample into a platinum crucible and ignite to constant weight.

Acidity or Alkalinity

Determine by colorimetric methods the pH of a 1% solution.

No. 37 GLUCOSE

$C_6H_{12}O_6$

Strength Determination

Weigh out 1 gm. of the sample, dissolve in water and dilute to exactly 250 cc. Then by means of pipettes transfer to a beaker 25 cc. each of Fehling's Solutions 1 and 2 and 50 cc. of the glucose solution. Bring this solution to a boil in 4 minutes and keep boiling for 2 minutes. Then filter through a weighed Gooch which has been washed with alcohol and ether and dried at 100°C. Wash the precipitate, first with warm water, then with alcohol, and finally with ether. Dry in the oven at 100°C., cool and weigh Cu_2O. For calculations see Molinari's Industrial Organic Chemistry, 2nd Edition, 1923, Page 582.

Ash

Ignite 1 gm. of the sample in a platinum crucible.

Average Analysis

Glucose 35%
Ash .....not over 0.2%

No. 38 GLYCERINE

$CH_2OH.CHOH.CH_2OH$

Specific Gravity

Determine by hydrometer at 15°C.

Ash

Ignite about 1 gm. in a platinum crucible.

Anhydrous Glycerine

Acetin Method.

1 to 1.5 gms. of glycerol, 3 gms. of anhydrous sodium acetate and 7 cc. of acetic anhydride are heated in a reflux apparatus for 1½ hours, then cooled and 50 cc. water slowly added through the condenser tube. The solution is warmed until the oily deposit dissolves, then filter and wash. The filtrate when cold is just neutralized with N/2 caustic soda solution using Phenolphthalein as an indicator. This operation is done slowly with constant agitation to avoid a local excess of caustic. 25 cc. of a 10% pure caustic soda solution is now added from a pipette and the mixture boiled for 15 minutes. Then the excess of alkali is titrated with N/1 HCl. Meanwhile 25 cc. of the same 10% soda solution is also titrated with N/1 HCl. The difference in these two titrations is calculated to glycerol.

1 cc. of normal HCl = 0.03067 gms. glycerol

Average Analysis of Dynamite Glycerine

Specific Gravity 1.252
Anhydrous Glycerol 95.12%
Ash .....None

No. 39 GUM SUDS

(Boil-off Liquor)

Adaptation of the Micro-Kjeldahl Method for Nitrogen Determination to Gum Suds Analysis.

1. Reagents: (a) A 30% solution of hydrogen peroxide (e.g. Merck's Superoxol). (b) A 1:1 solution of sulfuric acid. (c) The modified Nessler-Folin reagent, prepared as follows:

Dissolve 22.5 grams of iodine in 20 cc. of water containing 30 grams of potassium iodide. After the solution is complete, add thirty grams of pure metallic mercury and shake the mixture well, keeping it from becoming hot by immersing it in tap water from time to time. Continue this until the supernatant liquid has lost all of the yellow color due to iodine. Decant the supernatant aqueous solution and test a portion by adding a few drops thereof to 1 cc. of a 1% starch solution. Unless the starch test for iodine is obtained, the solution may contain mercurous compounds. To the remaining solution add a few drops of iodine solution of the same concentration as above, until a faint excess of free iodine can be detected by adding a few drops thereof to a 1 cc. volume of starch solution. Dilute to 200 cc. and mix well. To 975 cc. of an accurately prepared 10% NaOH solution now add the entire solution of potassium mercuric iodide prepared above. Mix thoroughly and allow to clear by standing.

(d) Stock solution of ammonium sulfate, prepared as follows:

4.773 gms. $(NH_4)_2SO_4$ dissolved in N/20 H_2SO_4 and made up to one liter with this acid. 1 cc. of this stock solution = 0.00001 gm. nitrogen.

2. Procedure.

Pipette 1 cc. of gum suds into a pyrex test tube and add 1 cc. of 1:1 sulfuric acid. Digest this over a low gas flame or at a low temperature on the electric heating unit. A few bits of pulverized glass should be added to lessen the tendency to bump. In case of the latter mode of heating, occasional agitation by hand is more or less essential, while in the former case continual shaking is necessary.

When the contents of the tube become charred and white fumes are given off, it should be cooled about one minute. Add from three to ten drops of 30% H_2O_2 (superoxol), agitating gently, and heating at a low temperature. If the contents of the tube become colorless, continue the heating from three to four minutes, at the end of which time it may be considered digested if no color returns.

In most cases several applications of superoxol and repeated heating are necessary to decolorize the gum suds. Occasionally it is even necessary to add another cubic centimeter or so of sulfuric acid, but this is exceptional and usually occurs only in gum suds especially rich in soap.

Dilute the digested liquor to 100 cc., mix thoroughly and pipette an aliquot part for Nesslerization. About 5 cc. usually gives a color sufficiently deep for comparison, but any suitable volume may be used. To the Nessler tube containing this aliquot part, add about 20 cc. of distilled water and 12 cc. of the special Nessler reagent. Dilute to 50 cc. and compare with the standards.

The standards are made from the stock ammonium sulfate solution, the nitrogen content of which has been previously checked by the usual Kjeldahl process. For example, from 0.6 to 1.6 cc. of the stock solution (in any number of steps desired) are mixed with 1 cc. of 1:1 sulfuric acid and diluted to about 30 cc. volume. 12 cc. of the special Nessler reagent are added and the whole diluted to 50 cc. total volume.

Care should be taken to keep the alkalinity of the standards and of the unknown about the same, because variations in alkalinity affect the intensity of the color.

No. 40 GUM ARABIC

Moisture

Dry about two grams of the sample to a constant weight at 100°C.

Quantitative Estimation

Fifty gms. of copper acetate are dissolved in water, an excess of ammonia added and the solution diluted to a liter with water and alcohol in such proportions that the final solution contains 50% alcohol. Dissolve 5 gms. of gum arabic in a liter, pipette out a 50 cc. aliquot and add 50 cc. of alcohol and 25 cc. of the copper reagent with constant stirring. Allow the precipitate to settle, then filter on a gooch, wash with 50% alcohol containing a little ammonia and dry to constant weight at 105°C. After weighing, ignite and weigh the ash. The difference in the two weights represents gum arabic.

Ash

Burn one gm. of the sample as usual in a weighed porcelain crucible.

Viscosity

Dissolve 25 gms. of the sample in warm water, dilute to 250 cc. and ascertain the time required to empty a 100 cc. pipette of this solution at a temperature of 30°C.

Impurities

Test with an iodine solution for starch and dextrine and with Fehling's solution for any reducing sugars.

Average Analysis

Gum Arabic	90%
Viscosity	23 seconds
Moisture	13.5%
Ash	3.0%

No. 41 HYDROGEN PEROXIDE
(Albone)
H_2O_2

Strength Determination

Measure out 2 cc. with a pipette into a beaker, dilute and add 15 cc. of dilute sulfuric acid. Titrate with N/10 potassium permanganate until a faint pink color is perceptible. Calculate the volume of oxygen (O_2) corresponding to 1 cc. of hydrogen peroxide. 1 liter of oxygen weighs 1.43 gms.

1 cc. N/10 $KMnO_4$ = 0.0016 gms. oxygen

Free Acid

Pipette out 25 cc., dilute and titrate with N/10 NaOH using Methyl Red as an indicator. Calculate as H_2SO_4.

1 cc. N/10 NaOH = 0.0049 gms. H_2SO_4.

Residue

Pipette 100 cc. of the sample into a weighed platinum evaporating dish and evaporate to dryness on the steam bath. Ignite at low red heat, cool and weigh.

Average Analysis of Albone

Volumes of oxygen	25
Free acid as H_2SO_4	0.4%
Residue	4.9%

No. 42 INDIGO CARMINE

Volumetric Determination

Weigh out about 10 gms. of the sample and dilute to a liter in a volumetric flask. Take a 50 cc. aliquot, add 20 cc. of dilute sulfuric acid, and 150 cc. of water. Titrate with N/50 permanganate until the greenish liquid changes to a light yellow. Calculate as indigotin.

1 cc. N/50 $KMnO_4$ = 0.0015 gm. indigotin

Free Acid

Pipette out 100 cc. of the above solution and add 32 gms. of neutral sodium chloride. Let stand for an hour with occasional stirring, then filter and wash with a little saturated solution of salt. Titrate the filtrate with N/10 NaOH using Phenolphthalein as an indicator. Calculate as sulfuric acid.

1 cc. N/10 NaOH = 0.0049 gms. H_2SO_4.

Average Analysis

Indigotin	6.5%
Free Acid as H_2SO_4	22.5%

No. 43 INDIGOTIN

Volumetric Determination

Dissolve 2.5 gms. of the sample in a liter of water, take a 50 cc. aliquot and proceed as in indigo carmine.

Free Acid

Use 100 cc. of the above solution and proceed as in Indigo Carmine.

To be concluded in the next issue of the Reporter.

Chemical Tests in the Wool Industry*

Methods Employed by Wool Industries Research Association

By **EDGAR HILL, F.I.C., A.R.C.Sc.**
(*Wool Industries Research Association*)

It is proposed in this paper to review briefly the various chemical tests relative to wool and its manufacture, these tests being employed in the laboratories of the Wool Industries Research Association.

IF we begin with a consideration of raw wool, we find that it consists of:—

	Per cent.
Wool Fiber	15 to 72
Yolk (Grease and suint)	12 to 47
Vegetable matter, dirt, etc	3 to 24
Moisture	4 to 24

The above constituents are tested for as follows:—

The wool is spread out in the humidity room kept at a standard humidity and after twenty-four hours is ready for sampling.

Moisture is determined in a specially designed bottle which is heated in an electric oven.

Grease is estimated by extracting 20 grams of dried wool with dry benzene in an all glass soxhlet apparatus. The extracted grease is examined for traces of suint, which are removed by washing a petroleum ether solution of the extracted grease with 50 per cent. alcohol.

Suint is removed from the benzene extracted wool by soxhlet extraction with water, and the percentage of suint is determined from the dried aqueous extract plus the trace of suint obtained from the grease.

Wool and Dirt.—The extracted wool is dried and weighed, then washed in warm soapy water and finally in plenty of warm water to get rid of dirt, care being taken to avoid fiber loss. The vegetable matter is hand picked from the wool and the clean wool is then dried and weighed, the dirt being estimated by difference.

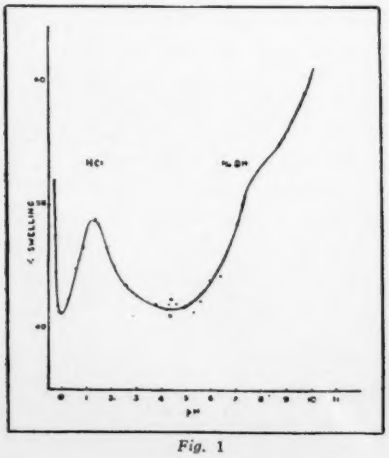


Fig. 1

We have now arrived at the stage when it will be advisable to consider some properties of wool. Microscopic examination shows the wool fibers to consist of a thin external layer of scales adhering to a solid central cortex which constitutes the main body of the wool. Chemical analysis reveals wool to contain carbon, hydrogen, oxygen, nitrogen and sulphur, together with a small amount of inorganic matter. Wool protein or keratin is of a complex but unknown composition, but when it is treated with certain hydrolyzing agents, which open up the protein molecule by the addition of water molecules, a number of substances result. The chemical composition of these hydrolysis products is known. They are found to be amino acids which therefore possess amphoteric properties, i.e., uniting with acids in acid solution and with alkali in alkaline solution. Wool possesses amphoteric properties and acid or alkali taken up by the wool has a great influence on its swelling, power of attracting moisture and its behavior throughout the various processes from the raw material to the finished article. Fig. 1 shows the swelling curve of wool in acid and alkaline solution.

Estimation of Alkali in Wool.—We are now in a position to consider the testing of wool for alkali taken up during wool washing, scouring and milling processes. On the basis of the amphoteric properties of wool, Hirst and King[1] have devised a method for the estimation of alkali in wool. Terephthalic acid, which is a stronger acid than soap acids but which has a very low solubility, is used (at 20 deg. C., 1 liter of water dissolves only 0.0168 grm. of this acid, less than 2/1000 per cent.) The sodium salt, however, is very soluble in water and is not decomposed by CO_2.

On adding excess of this acid to wool, the alkali present is converted into the soluble sodium salt of the acid; the wool becomes acid, and the sodium terephthalate is then free to diffuse through the solution. The terephthalic acid is used merely as a vehicle for the extraction of alkali from the wool and the amount of this acid taken does not enter into the calculation. Full details are given in the appendix.

Indicators have been found to be very useful for the

*Paper read at the Textile Institute Annual Conference on May 20th.

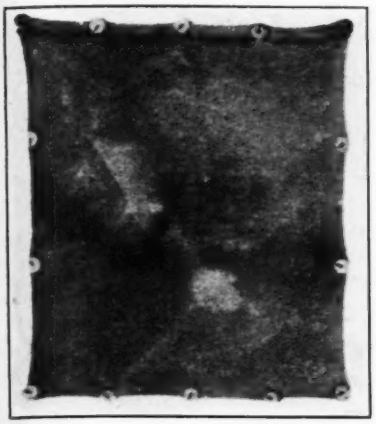


Fig. 2

testing of cloth for alkalinity. King[2] utilized indicator dyed cloth for the investigation of the migration of alkali to cuttle edges of pieces during the drying of moist cuttled piece goods and for uneven distribution of alkali in pieces, as shown in Figs. 2 and 3.

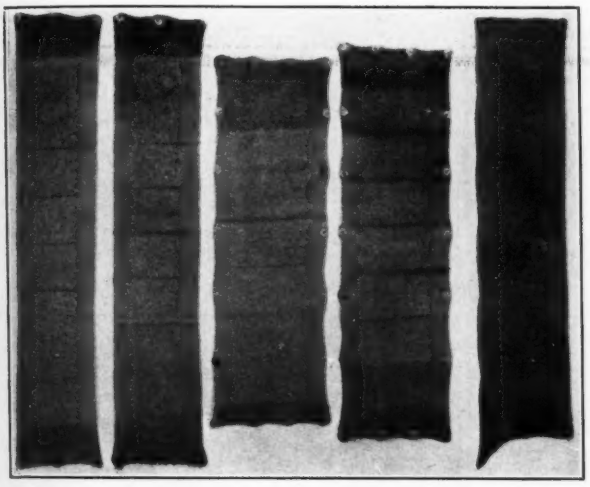


Fig. 3

Effect of Alkali on Dyes

The presence of alkali in wool is found to have an effect on dyestuffs, and King[3] showed that certain azo-dyes which normally are unaffected by sulphur dioxide in the presence of acid are seriously affected if the wool or yarn dyed material is alkaline when finished. The extent of the change depends upon the amount of alkali in the wool, since the more alkaline the wool, the greater is the amount of sodium hydrogen sulphite formed, with the consequent production of the bisulphite compound of the dye. Fig. 4 indicates the percentage of dyestuff in N/50 solution.

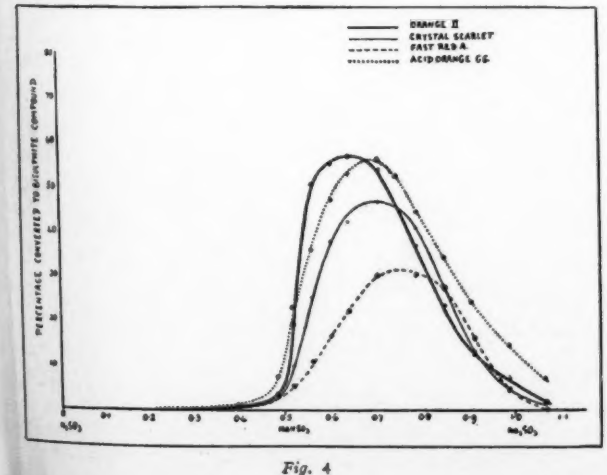


Fig. 4

With sulphur dioxide (H_2SO_3) alone there is no reaction in the acid range, but as the percentage of alkali increases we see there is a range about pH7 where the maximum formation of the bisulphite compound occurs. These compounds possess a different shade from the original dyestuff, are more soluble and fade much more easily than the original dye. As a result of this work King[17] has produced a new stoving test for dyestuffs, which is described in the appendix.

Estimation of Soap in Wool[18].—When an unknown sample is submitted for analysis, it is first necessary to extract with petroleum ether to remove oil as such. This oil should be examined for soap and if present should be determined. The wool is then extracted with alcohol to remove the soap, and it is found that extraction with alcohol gives excellent results. Before use the alcohol should be re-distilled and neutralized and all operations should take place in glass vessels.

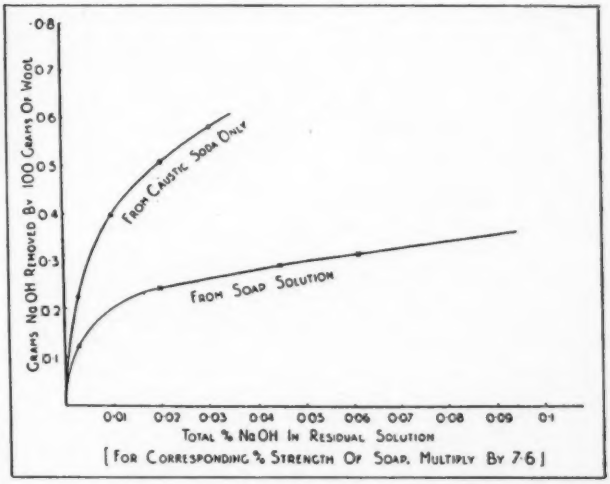


Fig. 5

Since neutral soap is "caustic" to wool and as scouring with neutral soap alone is not practiced in the majority of scouring processes, there will be obviously an absorption of alkali by the wool. Fig. 5 is a graph of the results obtained, showing the absorptive effects of caustic soda and soap solution. A similar curve for sodium carbonate would presumably lie between the two curves in Fig. 5.

Oil Testing.—The usual standard methods of oil analysis are employed, but it may be of interest to note that for the determination of iodine values, although Wij's solution is quite satisfactory for routine work, in spe-

cial cases where oxidation has occurred or in the case of wool grease, it is preferable to use Hubl's solution, for the following reasons. Holde[4] found on using Hubl solution, that Cholesterol gave an iodine value of 73 to 77 (65.7 theoretical) and Phytosterol 41 to 76 according to the time given for the reaction, but with Wij's they gave the value of 135 for both, which is approximately double the theoretical value. McLean and Thomas[5] found that the acid in Wij's solution acts in certain cases as an enolyzing agent on carbonyl compounds and that the addition of halogen then takes place. The Hubl agent, being neutral, has little or no enolizing action.

The effect of ultra-violet light and the use of the fadeometer have been found by Hirst[6] to be of service for testing both samples of oils and pieces for spots of oil, and also to account for the development of stains in dyeing.

A highly unsaturated oil or one containing unsaturated impurities shows, under ultra-violet light, an intense fluorescence on the surface of the oil, the deep penetration of the light being prevented by the screening effect of the unsaturated material. If the oil be diluted with non-fluorescing purified paraffin or petroleum ether, a value may be obtained by dilution at which the fluorescence extends downwards throughout the whole of the oil.

Mineral Oils.—Give a yellow to brown stain when subjected to sunlight or to the fadeometer and show with ultra-violet light a fluorescence from blue to white respectively. Highly purified mineral oil such as medicinal paraffin gives no stain on exposure to sunlight or to the fadeometer, and shows no fluorescence with ultra-violet light. A typical American spindle oil gave a blue fluorescence and on being exposed for 175 hours in a quartz tube to the fadeometer, a brown precipitate formed which was insoluble in petroleum ether, while the fluorescence was then seen to have changed to white.

Spots of Mineral Oil on cloth show a blue fluorescence, and on being submitted to sunlight or to the fadeometer or for a much shorter time to steaming in air, oxidation takes place. On subjecting the cloth to a light scour and then dyeing, it is found the spot now appears to be darker in shade than the remainder of the piece, indicating a greater penetration of dyestuff or increased dyeing affinity at the spot.

Vegetable Oils.—Give varying shades of fluorescence from blue to yellow but usually a blue. Cotton seed oil after twelve days' exposure in a quartz tube to the fadeometer gave a yellow fluorescence and after six weeks, no fluorescence. As would be expected, the iodine value of the oil decreases with time of exposure and the viscosity of the oil increases.

Spots of Vegetable Oil on wool do not exhibit pronounced fluorescence because this is similar to the fluorescence of the wool. On being exposed to sunlight or the fadeometer or for a shorter time to the action of steam in air, oxidation occurs and the spots resist the action of a light scour. On dyeing the cloth, the spots appear to be of a lighter shade than the remainder of the piece.

Olive oil has been found to take the longest time of the vegetable oils tested before it resists to scouring and dyeing. Particulars of the scouring and dyeing processes are contained in the appendix.

Estimating Harsh Treatment

Harsh Treatment of Wool in Process Work.—Harsh treatment in process work is detected by the Pauly Test which has been developed by Rimington and Burgess[7] for the quantitative estimation of rough treatment or damage. Pauly utilized the fact that portions of the wool fiber, namely the cortex below the epithelium scales, are capable of coupling with diazotized sulphanilic acid with the formation of a brown color while the scales are unaffected. The test is in fact Erlich's well known diazo reaction for Phenols, Hydroxy Aromatic Acids and Imidazols.

Sieber[8] employed Benzopurpurine 10B for this purpose and found it unnecessary to diazotize the dye.

Kronacher and Lodemann[9] use Methylene Blue, while Herzog[10] uses a cold saturated solution of indigo-carmine, and to emphasize the color, he then stains the scales with picric acid.

Determining Sulphur

Sulphur in Wool.—There are two chief methods for the determination of sulphur in wool. These are based on the oxidation of sulphur to sulphate and the simultaneous destruction of the organic matter. The methods are as follows (1) The Carius Method. This method is found to be excellent, the chief disadvantages being the labor in sealing the tubes and the cost. (2) The Benedict Denis Method. This process is rapid but there is a danger of losing material during the ignition and not only is it necessary to use high-class chemicals, but also duplicate blank determinations should be made on the reagents. Both methods are described in the appendix.

Table 1 gives typical figures obtained by Barritt and Rimington[11]:

Estimation of Total Nitrogen.—The Kjeldahl method is employed and found to be quite satisfactory for nitrogen estimation.

Table 1

Sample	Total S by Carius method Per cent. dry weight.	Total S by Benedict Denis method Per cent. dry weight
Crossbred 50's wool	3.46	3.47
Turkey Mohair (coarse).	3.03	3.02
White Alpaca	3.92	3.85
Crossbred 40's wool	3.02	3.03

Barritt[12] obtained the following typical figures in dry wool.

Australian Merino 58's, 16.63 per cent; Australian Merino 60's, 16.78 per cent; Australian Merino 64's,

16.63 per cent; Welsh Mountain, 16.54 per cent; Alpaca White 17.00 per cent; Alpaca Brown, 16.66 per cent. and Alpaca Black, 15.85 per cent.

Effect of Alkaline Scour on Nitrogen Content.—1 per cent. sodium carbonate scour on New Zealand Wool caused an increase in the nitrogen content of 0.25 per cent. calculated on the wool or an increase of 1.50 per cent. of the total nitrogen.

2 per cent. sodium carbonate scour on Black Alpaca increased the nitrogen content by 0.37 per cent. calculated on the wool or 2.33 per cent. of the total nitrogen.

This indicates the removal of a soluble portion of lower nitrogen content than the wool by this alkaline treatment.

Effect of Carbonizing

Effect of Carbonizing on Nitrogen Content.—Two samples of burry wool carbonized with 10 per cent. sulphuric acid showed a decrease in nitrogen content of 0.20 per cent. and 0.22 per cent. respectively calculated on the wool or a 1.20 per cent. and 1.31 per cent. loss of nitrogen.

Effect of Chlorination on Nitrogen Content.—Barritt also treated worsted cloth with Chlorine Water and estimated the chlorine taken up by the wool and the percentage of nitrogen. The following figures were obtained:

Table 2

Percentage Chlorine taken up by the cloth	Loss of N calculated on cloth Per cent.	Loss of Total Nitrogen. Per cent.
3.53	0.04	0.24
6.89	0.18	1.09
10.26	0.23	1.39

Inorganic Matter.—Wool is difficult to ash on account of the fact that it swells up and froths under the effect of heat.

I have found the use of a saturated solution of ammonium nitrate in 67 per cent. alcohol for wetting the wool, to assist the combustion and to promote the formation of a porous carbonaceous mass. The burning off is conducted in the fume cupboard and the crucible is finally heated in the muffle.

A successful process for dissolving the wool prior to ashing, is to use hydrochloric acid as in the Benedict Denis method. Twenty c.c. of a 20 per cent. solution of ammonium nitrate solution are added, the solution evaporated to dryness, heating in the fume cupboard, and the fixed carbon finally burnt off.

Estimation of Metals in Ash.—The question of inorganic chemical and micro-chemical analysis here arises. I propose to summarize by stating that in addition to ordinary analytical methods employed, the micro methods of Pregl[19] and of Feigl[20] are used when necessary. The hydrogen electrode is used for checking hydrogen ion determinations and for the micro analysis of such irons as the determination of Calcium ions, and of Chlorine ions, and the use of the glass electrode is being investigated.

It may be of interest to conclude by indicating the lines on which chemical testing is being investigated.

Copper, Iron and Manganese

Copper, Iron and Manganese promote oxidation of organic matter, causing discoloration stains and spoil first class bleaching. The use of sulphocyanate for the detection of iron has generally met with the greatest response in laboratories.

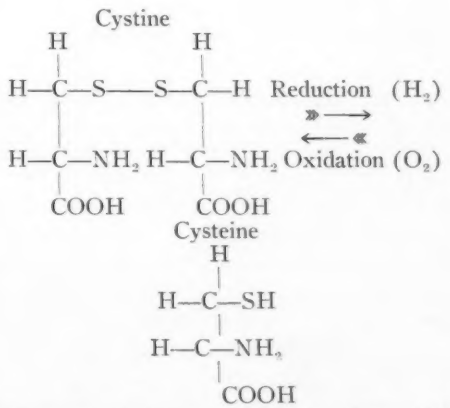
The red color with ferric salt is due to undissociated salt and its double compounds, the ionized salts are colorless, and the reaction is interfered with to a certain degree by fluorides, phosphates, arsenates, oxalates, citrates, tartrates and iodates, and to a less degree by acetates and sulphates.

Feigl[20] gives the recognizable limits with ferric iron.

Potassium Sulphocyanate 2.5×10^{-7} grm.
Potassium Ferrocyanide 5×10^{-8} grm.
Ferrous Iron
Dimethyl Glyoxime 4×10^{-8} grm.
size of drops 0.05 c.c.

Delicate Test for Copper and Iron

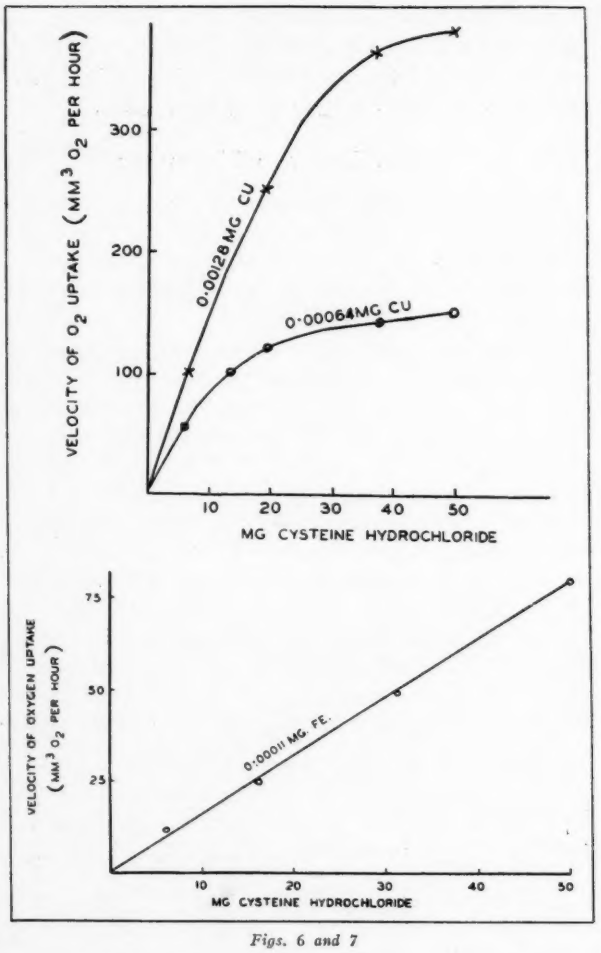
A most delicate method for the determination of minute traces of copper and iron has been employed by Warburg[13] who studied the catalytic effect of iron, copper and manganese on the rate of oxidation of Cysteine to Cystine.



By this method he succeeded in determining the amount of iron, copper and manganese in 1/10 cubic centimetre of blood serum, Elliott[14] obtained graphs (Figs. 6 and 7) showing the velocity of oxygen uptake in the presence of small amount of copper and iron respectively.

The catalytic effect of iron and manganese can be totally prevented by the addition of pyrophosphate which does not affect the activity of the copper.

As cystine exists in the keratin molecule in wool to the extent of about 3.1 per cent. and as free cystine is completely converted into cysteine by excess of sodium sulphite, the above reaction is being worked upon to see



Figs. 6 and 7

if there is any possibility if the micro analysis of iron, copper and manganese *in situ* on the wool.

Appendix

Moisture Determination.—Moisture is estimated by the use of a special bottle (Fig. 8).[21] One position of the tap enables a current of air to be drawn through the wool, the air entering at the bottom of the bottle. On turning the tap through 90 deg. the bottle is completely closed. The bottle containing the weighted wool sample (about 1 grm.) is heated in an electric oven at 104 deg. to 106 deg. C., and a current of dry air, dried by means of the usual drying agents (sulphuric acid and calcium chloride) drawn through.

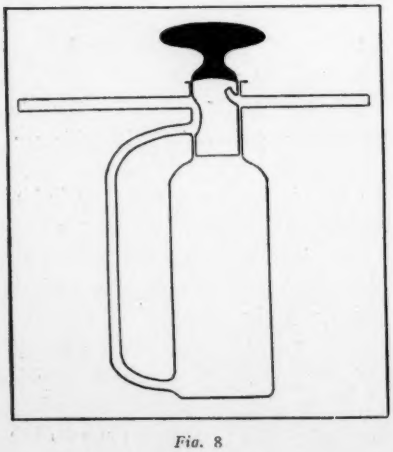


Fig. 8

After 1½ to 2 hours the tap is closed and the bottle then transferred to a desiccator for half-an-hour and then weighed. The bottle is re-heated for half-an-hour and its weight taken. This is repeated until the weight is constant and in almost all cases two re-heatings are sufficient.

Estimation of Wool Dirt, Grease and Suint.—The method outlined below is that according to Sutton,[15] but slightly modified.

The wool is spread out in a room of fairly uniform temperature and humidity, and after 24 hours is sampled. Two grm. samples are used for the determination of moisture content.

For the grease determination 20 grm. are taken and dried at 105 deg. C. to a constant weight. The sample is then transferred to an extraction thimble (43 mm. × 123 mm.) care being taken not to pack the wool too tightly. The extraction apparatus is insulated by means of asbestos tape and the extraction carried out with dry benzene for six hours. The benzene is evaporated off and the grease dried at 106 deg. C. and weighed.

This grease should then be examined for complete freedom from suint by burning a portion and testing for an alkaline ash. If this is present a known quantity of the grease is taken, dissolved in petroleum ether and washed several times with 50 per cent. alcohol in order to separate the suint from the grease. The purified grease obtained by distilling off the petroleum ether is then dried and weighed. If desired, the trace of suint may be recovered by distilling off the alcoholic washings, drying, and weighing.

The wool after extraction of the grease is gently dried in the thimble to get rid of benzene. It is then soxhlet extracted with water for six hours, the extract in the flask being taken to dryness and weighed. This residue is the suint.

The extracted sample of wool is then removed from the thimble, rinsed first in warm water, then in warm soapy water and finally in plenty of warm water, any foreign matter being picked out. After air-drying the clean sample is dried at 105 deg. C. and weighed. The weight of soil, seeds and other matter is obtained by difference.

Estimation of Alkali in Wool.—Take 10 grm. of wool to 200 c.c. of water, warm and add an excess of terephthalic acid, usually 0.4 grm., raise the temperature to 60 deg. C., stir and allow to stand for two hours. Pipette off 50 c.c. into a beaker and add 15 c.c. of N/10 acid to decompose the sodium terephthalate, filter off the terephthalate acid from the cold solution and wash with cold water collecting the filtrate and washings in a conical flask. Titrate the filtrate and washings with N/10 sodium hydrate using phenolphthalein or phenol red as indicator. Multiply the difference of the alkali used from the acid added by four. The result is equivalent to the amount of alkali in the wool.

The correction for the slight solubility of terephthalic acid, namely 0.0168 grm. per litre, is only necessary when the volume of the washings becomes excessive.

Estimation of Mineral Acid in Wool.—Take 10 grm. of wool to about 100 c.c. of water, warm and add an excess of sodium terephthalate, usually 50 c.c. of N/10, make up to 200 c.c. raise the temperature to 60 deg. C., stir and allow to stand for three hours. Pipette off 50 c.c. into a beaker and add 15 c.c. of N/10 acid to decompose the sodium terephthalate. Filter off the terephthalic acid from the cold solution and wash with cold water collecting the filtrate and washings in a conical flask. Titrate the filtrate and washings with N/10 sodium hydrate using phenol red or phenolphthalein as indicator. Multiply the difference of the alkali used, from the acid added, by four. This result is then equivalent to the amount of undecomposed N/10 sodium terephthalate originally added and the undecomposed N/10 sodium terephthalate found is equivalent to the acid contained in the wool.

Estimating Soap

Estimation of Soap in Wool.[18] — The alcohol used should be redistilled and neutral and preferably absolute. The material for analysis should be placed in a soxhlet extractor of 200 cc. capacity. 10 gram. of top or worsted silver or 26 grm. of 9 oz. worsted cloth are used. All operations should take place in glass vessels without corks.

Preparation: The dried sample is extracted in the soxhlet with petroleum ether to remove oil. This oil should be examined for soap, which if present should be determined by extraction with 50 per cent alcohol as in suint analysis.

Temperature: It is necessary to maintain the temperature of the alcohol and wool in the extractor at or near boiling point. To secure this the extractor is covered with asbestos paper or tape.

Duration of Extraction: Almost complete extraction of the soap is obtained by filling and emptying the extractor twelve times with hot liquor; the time taken is five to six hours. For ordinary analysis, twelve extractions are sufficient.

Treatment of the Solution

Treatment of the Solution: The solvent is filtered hot, before evaporation, through a thin Swedish filter paper No. 1.F. The bulk of the alcohol is distilled off, leaving about 50 c.c. This is poured into a wide mouthed flask of 100 c.c. capacity and the distilling flask is washed into the same. The alcohol is then evaporated to dryness on a water bath, and the residue dried in a steam oven, cooled in a desiccator under diminished pressure and weighed until constant. The extract is boiled with 60 c.c. distilled water, methyl orange or methyl red being added. A known excess of N/10 acid is then run in (25-30 c.c.), boiled and allowed to cool, and shaken out with ether three times. The ether must be neutral and is made so by re-distilling over lime. The ether is washed with water and the water added to the liquor. On evaporation the ether extract gives the total oil and fatty acid in the alcohol extract.

Fatty Acid: Dissolve the oil thus obtained in absolute alcohol or alcohol and ether, add a few drops of a 1 per cent solution of phenolphthalein in alcohol and titrate with a tenth normal aqueous alkali. The end point is a pink color which lasts two minutes, though after that it will probably disappear.

In the case of difficulty in seeing the end point owing to the darkness of the solution it may be necessary to adopt the method of Bodtker who shakes the oil with 25 c.c. of alcohol, 50 of water, sufficient N/10 soda to give a strong red color with phenolphthalein and then titrates back. In each case also a fluorescent dye such as Eosin can be used as an indicator.

Soap: The residual liquor after shaking with ether is titrated back with N/10 NaOH and the difference between added acid and back titration is calculated as sodium oleate.

Determining Calcium Soaps

Calcium: The calcium soaps are included above as sodium oleate, but may be determined as follows:—The liquor after shaking with ether and back titration for soap is evaporated to dryness, washed with a little ether and dissolved in water. Ammonia, ammonium chloride and ammonium oxalate are added and the mixture allowed to stand for several hours. The precipitate is filtered, washed and dissolved in warm 2 per cent sulphuric acid, then titrated hot with N/10 potassium permanganate. From this the amount of calcium is calculated and expressed as calcium oleate (or sodium oleate). With one set of extractions 0.09 per cent calcium soap has been obtained.

New Standard Test for Fastness to Stoving.—King[17] makes use of cloth dyed with Bromthymol Blue which he uses as indicator cloth, and he carried out the test as follows:—While the dye under test is being applied to the cloth, a similar pattern of Bromthymol Blue indicator cloth is treated in an acid bath under the same conditions with the exception that the dye is omitted. The two patterns are washed off together to leave the same residual acidity. They are then treated together in the soap solution until the yellow indicator cloth changes to a greenish blue shade, preferably matched against a standard, otherwise there is a little uncertainty as to alkalinity of the patterns.

The pattern under test is now divided into two portions, and these, along with the indicator cloth pattern are hung in the stoving vessel. One portion is removed immediately the indicator cloth pattern has become just yellow (which requires a minute or so), put aside under a bell jar over water to keep it damp, and examined along with the other portion when this is removed at the end of the stoving period. The fastness is expressed thus:—

Ordinary Test	Alteration	Bleeding
New Test	Alteration	Bleeding

Wool Scour for Oil Tests and Dyeing Particular.—Scour for 20 minutes in 0.2 per cent potash olive oil soap and 0.1 per cent soda ash, 50 c.c. per grm. of cloth at 45 deg. C. with constant agitation in thermos flask. Wash in running water. Dye with 0.5 per cent Fast Blue RH, and 5 per cent of 50 per cent acetic acid.

Test for Harsh Treatment

Test for Harsh Treatment in Process Work.—Solutions required—Sodium Sulphanilate 10 per cent: 88.7 grm. of sulphanilic acid dissolved in one litre of water containing 20.5 grm. of sodium hydroxide. Sodium Nitrite 8 per cent: 80 grm. dissolved in one litre. Sodium Carbonate 9 per cent: 243 grm. of the crystalline salt in 1 litre. Sodium Hydroxide 10 per cent: 100 grm. dissolved in water and made up to 1 litre. "New Acid Brown S" 0.1 per cent: 1 grm. of dyestuff dissolved in 1 litre of water and the solution filtered. Concentrated Hydrochloric Acid: Density 1.16.

The reagent is prepared by mixing 10 c.c. of a 10 per cent solution of Sodium sulphanilate with 5 c.c. of an 8 per cent solution of sodium nitrite, then adding 2 c.c. of concentrated hydrochloric acid down the side of the vessel mixing with a gentle motion and allowing to stand for one minute before use. A sample of the material is taken weighing about 0.1 or 0.2 grm. (greasy wool should be washed first in benzene or ether) and wetted out in 15 c.c. of a 9 per cent sodium carbonate solution, and to this the reagent is added. After exactly 10 minutes the wool is withdrawn, rinsed thoroughly in water and transferred to a test tube. 4 c.c. of 10 per cent sodium hydroxide solution are then added and the tube placed in a bath of boiling water for exactly 5 minutes.

Colorimeter Measurements

The tube is cooled under the tap and the resulting reddish solution transferred to one ("Wool") of two similar graduated measuring cylinders, marked "Standard" and "Wool" respectively. Water is added, rinsing out the test tube to bring the volume up to 25 c.c. In order to compare the strength of this solution with the standard (0.1 per cent New Acid Brown) it is necessary to use a Dubosc or Rober colorimeter, or else the cylinders are placed side by side on a sheet of white paper near to a window and sufficient standard dye solution is poured into the cylinder marked "Standard" until, when viewed from above, the color appears identical in the two.

When a match is obtained the volume of solution in the "Standard" cylinder is recorded.

Construction of the Numerical Scale.—For convenience it has been decided to assign the value of "100 units of damage" to a sample of wool (or cloth), such that 0.1 gram treated as above and made up to 25 c.c. final volume gives a solution of color intensity exactly equal to that of the standard dyestuff (0.1 per cent). Values are then calculated for individual samples as may be seen from the example worked out below.

Weight of sample taken 0.100 grm.

Total volume of final solution, 25 c.c.

The standard dyestuff solution set at 13.7 matched the wool solution set at 25.

The number of units of damage for the sample is therefore

$$\frac{13.7}{25} \times 100 = 54.8$$

A convenient formula is obtained as follows:

Let the volume of solution in the "Standard" cylinder be S. and W. = the weight of wool taken.

$$\text{Damage} = 100 \times \frac{S}{25} \times \frac{0.1}{W}$$

$$= \frac{S}{W} \times 0.4 \text{ units.}$$

Determining Nitrogen

Determination of Nitrogen.—For each determination two samples of wool are taken. One sample about 0.7 grm. in weight for the Kjeldahl digestion and about 1 gram for the determination of moisture content.

The Kjeldahl method employed is as follows: (J. Barritt[12]). The sample is heated gently for about an hour with 20 c.c. of nitrogen free sulphuric acid, and the flask allowed to cool; 10 grm. of potassium sulphate are added and the contents of the flask boiled until colorless, the heating being continued for at least 1 hour. The cold liquid is then poured into 100 c.c. of cold water contained in the distilling flask of an ammonia distillation apparatus, and the flask rinsed with successive small quantities of water, which are also transferred to the distilling flask.

The apparatus used for the ammonia distillation is that described by Stock[16] with minor modifications; 25 c.c. approximately 0.5N sulphuric are distributed between the receiver flask and the attached Peligot tube, the acid in the Peligot tube being colored by the addition of two drops of methyl red. A few pieces of pure zinc foil are dropped in the distilling flask, to the contents of which a few drops of methyl red are added. Strong caustic soda solution is run in from a funnel, care being taken that the liquid seal is not broken so as to ensure that no gas escapes.

The time of distillation is of the order of 1½ hours and the contents of the flask and Peligot tube being finally washed out into a 600 c.c. baker and titrated with approximately 0.1N sodium hydroxide, the end point being very sharp.

Duplicate determinations agree usually to within 1 in 150 and are often more concordant, and a blank determination carried out using 1 grm. of dextrose gave a result affecting the determination to less than 1 part in 400 and was therefore neglected. The results obtained on the wet weight of the wool are reduced to the standard basis of dry weight from the determination of moisture content made on the control sample.

Estimation of Organic Sulphur.—Preparation of Material—The sample is treated two or three times with sulphur free benzene at about 45 deg. C. to 50 deg. C., air dried and then hand sorted to remove all small particles of foreign matter. It is re-washed in benzene, dried off, washed with 0.1 per cent saponin solution in distilled water, then washed with many changes of distilled water, dried and allowed to condition in a suitable room free from sulphur fumes and maintained at a definite relative humidity.

Carius Method

Weighing out of the Wool or Hair.—Samples of the order 0.25 to 0.5 grm. are weighed out for sulphur determination and a sample of about 1 grm. is taken for a moisture determination which is best carried out by the "Bottle" method of Barritt and King (supra), the principle of the method being to dry out the wool by heating to 104 deg. C. to 106 deg. C. in a current of dry air.

Determination of Sulphur.—(1) *Carius Method* (Barritt and King).[21]—About 3 c.c. of fuming nitric acid (S.G. = 1.53) are transferred by means of a long funnel and pipette to the bottom of a Carius tube, and the wool transferred by means of forceps and pushed down the tube to within about 6 inches of the bottom by means of a glass rod. The tube is then sealed in the usual manner, and estimations are carried out in duplicate for each wool. The tubes are heated together in a Carius furnace and in a period of about 1½ hours the tubes are raised to 250 deg. C. and maintained at this temperature for about 8 hours. Each tube is allowed to cool overnight and is opened up by softening the tip with a Bunsen flame, a pressure being found in the tube. After withdrawing from the furnace and cutting off the ends, the tubes are gently warmed to expel dissolved gases and the contents washed into beakers, evaporated to dryness (on steam) with the addition of hydrochloric acid, this being repeated to remove all traces of nitric acid, which is detrimental to the sulphate estimation.

Precipitation With Barium Chloride

About 100 c.c. of hot distilled water are added, and the contents filtered off to remove any trace of glass introduced when opening the tubes. The liquid, about 400 c.c. in volume, was acidified with dilute hydrochloric acid, heated to boiling and precipitated by the slow addition of 50 c.c. of boiling N/20 barium chloride solution. After keeping hot for some hours and allowing to stand overnight, the barium sulphate was collected and estimated on a weighed Gooch crucible in the usual way. It should be especially noted that to ensure complete breaking up of the wool it is essential that the temperature of the tubes be not less than 200 deg. C. and further that not only a short length, at most one inch, of tube project beyond the iron furnace tube.

(2) *Benedict Denis Method.* Rimington[11]. 10 c.c. of hydrochloric acid solution (2 parts concentrated acid, 1 part water) are pipetted into a silica crucible (40 c.c. size) which is then heated on the hot plate until the contents begin to simmer. The wool is added (0.2 to 0.5 grm.) using a glass rod to aid transference from the weighing bottle, and the mixture warmed with gentle stirring until solution is effected. 1 c.c. of Benedict Denis solution is then added and the mixture evaporated to dryness on the water-bath; a further 4 c.c. of reagent is added, followed by evaporation, after which the crucible is covered with a lid of the "peaked" or "platinum shaped" type. The contents are *carefully* ignited with a small flame directed first obliquely downwards on the lid thus heating the contents by reflux. A vigorous reaction occurs accompanied by some decrepitation and for this reason it is essential that the lid should be well fitting. When crackling has subsided, the full flame of the Bunsen is directed on to the bottom for 2 to 3 minutes and the crucible then allowed to cool.

Adhering Particles

The lid is removed and placed in a suitably-sized evaporating basin; 5 c.c. of 10 per cent hydrochloric acid solution are then added together with a little water and the basin is heated until its contents boil. Any particles adhering to the crucible lid are dissolved during the operation, the solution together with washings being transferred to a beaker. The open crucible is next heated to red heat for 10 minutes, allowed to cool, and the contents are dissolved (by heating on the water bath) in 10 c.c. of 10 per cent hydrochloric acid to which are added about 10 c.c. of water. No undissolved particles should remain: if specks of carbon are present, the operative procedure must have been at fault. Solutions and washings are transferred to the beaker: the combined liquids are filtered, and precipitation is accomplished by running in 25 c.c. of hot 10 per cent barium chloride solution, the main bulk of the liquid being maintained just short of boiling temperature. After keeping overnight, the precipitate is filtered, washed, and dried in the usual way.

Benedict Denis Solution.—25 grm. Copper Nitrate, 25 grm. Sodium Chloride, 10 grm. Ammonium Nitrate per 100 c.c. of solution.

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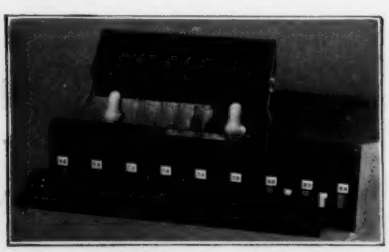
New Boiler Water Testing Equipment

W. A. Taylor and Company, Inc., 872 Linden Avenue, Baltimore, Maryland has recently developed and placed on the market two new outfits for controlling the pH and phosphate content of boiler water. Both outfits are similar in design and operation to the Slide Comparator shown in the illustration. They can be secured as separate units or in combination as described.

All color standards used in these sets, as well as those employed in all other Taylor equipment, are guaranteed by the manufacturer to retain their accuracy for a period of five years.

The pH outfit contains a new indicator thymol red (yellow 8.0-11.2 red) developed by the manufacturers. The color standard slide contains standards from 8.0-11.2 in intervals of 0.4 pH, giving sufficient accuracy for boiler water control.

The Phosphate Slide Comparator employs a method developed in the Taylor Laboratories with the purpose of eliminating errors due to constituents other than phosphate, such as silicate, iron, etc. Silicates as high as 1.0 percent as SiO_2 and ferric iron as high as 50 parts per million introduce no error. Ferrous iron does not interfere at all. The color standard slide contains standards for 5, 10, 20, 30, 40, 50, 60, 80 and 100 parts per million of phosphate as PO_4. Concentrations of phosphate up to 100 parts per million can be determined directly and higher concentrations can be taken care of by dilution. The reagents employed are very stable.



The procedure is so simple that anyone can quickly learn to use the outfit and only a few minutes are required to complete a test. The manufacturers will gladly send complete details on request.

New Development

A new development by which cotton fibres are chemically treated so that the fibres curl, producing a permanent wool-like texture, was demonstrated by a recent display of the Bellman Brook Bleachery in one of the Boardwalk windows at the Du Pont Exhibit, Atlantic City, N. J. A lamb made of cotton, a number of cotton bolls mounted on sprigs to simulate growing cotton and a model dressed in one of the fabrics were among the attractions. Several lengths of Dovelin, a Butterfield fabric, and Cameron print, an Arthur Bier fabric, were used as trimmings. Du Pont dyestuffs were featured in the display.

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The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

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